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DETERMINATION OF DIOXIN LEVELS IN CARBON REACTIVATION PROCESS EFFLUENT STREAMS

by

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16. ABSTRACT

A preliminary study was performed to evaluate the potential formation and persistence of tetrachlorodibenzo-p-dioxins (TCDDs) and tetrachlorodibenzo furans (TCDFs) in the effluent streams of a fluidized bed system used for thermal reactivation of granular activated carbon (GAC) that had been used for treating Cincinnati, Ohio municipal drinking water.

The total TCDD concentrations found in the stack emissions (particulate material and gaseous emissions) ranged from 0.06 to 0.3 nanograms per dry, standard cubic meter (ng/dscm). TCDDs were detected in the dryer off-gas cyclone catch samples from all four tests and in the scrubber water samples from two of the four tests. The total TCDD concentrations found in the cyclone catch ranged from 0.20 to 4.14 nanograms per gram (ng/g). TCDDs at concentrations of 0.12 and 0.21 nanograms per liter (ng/L) were found in the scrubber water from two tests.

TCDFs were detected in stack particulate samples from three tests and in the vapor phase (XAD-2) samples from two tests. The total TCDF concentrations found in the stack emissions (particulate material and gaseous emissions) ranged from 0.08 to 0.51 ng/dscm. TCDFs were detected in the dryer off-gas cyclone catch from all four tests at concentrations ranging from 0.48 to 3.33 ng total TCDFs/g.

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FOREWORD

The U.S. Environmental Protection Agency was created because of increasing public and government concern about the dangers of pollution to the health and welfare of the American people. Noxious air, foul water, and spoiled land are tragic testimonies to the deterioration of our natural environment. The complexity of that environment and the interplay of its components require a concentrated and integrated attack on the problem.

Research and development is that necessary first step in problem solution, and it involves defining the problem, measuring its impact, and searching for solutions. The Municipal Environmental Research Laboratory develops new and improved technology and systems to prevent, treat, and manage wastewater and solid and hazardous waste pollutant discharges from municipal and community sources, to preserve and treat public drinking water supplies, and to minimize the adverse economic, social, health, and aesthetic effects of pollution. This publication is one of the products of that research and is a most vital communications link between the researcher and the user community.

This report presents the results of a preliminary study to evaluate the potential formation of either tetrachlorodibenzo-p-dioxins (TCDDs) or tetrachlorodibenzo furans (TCDFs) during thermal reactivation of spent granular activated carbon. The study is part of an effort to assess the net environmental effects of water purification with granular activated carbon.

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ABSTRACT

A preliminary study was performed to evaluate the potential formation and persistence of tetrachlorodibenzo-p-dioxins (TCDDs) and tetrachlorodibenzo furans (TCDFs) in the effluent streams of a fluidized bed system used for thermal reactivation of granular activated carbon (GAC). The batch of carbon reactivated during the study had been used for approximately 1 year to treat Cincinnati, Ohio, municipal water.

Sampling of the reactivator stack gas and particulates, particulates from the dryer off-gas cyclone, scrubber and quench waters, and the spent carbon feed was conducted during four tests performed over a 3-day period. During one test, the natural gas was sampled with an XAD-2 trap to determine if polychlorinated biphenyls (PCBs) were present in the supply used to fire the carbon reactivation system. The stack effluent was sampled with an EPA Method 5 train that had been modified by adding a water-cooled XAD-2 trap between the filter and first impinger to collect TCDDs and TCDFs in the vapor phase of the sample stream. Grab and composite procedures were used to collect samples of the other effluent streams.

TCDD and TCDF analyses of the reactivator effluent samples and spent carbon feedstock were performed by Soxhlet or liquid-liquid extractions, removal of interferences with liquid chromatographic columns, and determination of the TCDDs and TCDFs by combined high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS). PCB analyses were performed by Soxhlet extraction of the XAD-2 followed by determination of the PCBs by combined high resolution gas chromatography/mass spectrometry (HRGC/MS).

TCDDs were detected in samples of the stack emissions, the particulate material collected by the dryer off-gas cyclone, and the scrubber water. TCDDs were not detected in any quench water or spent carbon feed samples from the four tests. TCDDs were detected in the stack particulate samples from all four tests and in the vapor phase (XAD-2) sample from one test. The total TCDD concentrations found in the stack emissions (particulate material and gaseous emissions) ranged from 0.06 to 0.3 nanograms per dry, standard cubic meter (ng/dscm) and emission factors ranged from 0.49 to 1.11 nanograms (ng) total TCDDs/kilogram (kg) of spent carbon feed. TCDDs were detected in the dryer off-gas cyclone catch samples from all four tests and in the scrubber water samples from two of the four tests. The total TCDD concentrations found in the cyclone catch ranged from 0.20 to 4.14 nanograms per gram (ng/g). TCDDs at concentrations of 0.12 and 0.21 nanograms per liter (ng/L) were found in the scrubber water from two tests. The isomers identified in the various effluent samples were: 1,3,6,8-TCDD, 1,3,7,8-TCDD, 1,3,7,9-TCDD and 2,3,7,8-TCDD.

TCDFs were detected in stack emission and dryer off-gas cyclone catch samples, but were not detected in any scrubber water, quench water, or spent carbon feed samples from the four tests. TCDFs were detected in stack particulate samples from three tests and in the vapor phase (XAD-2) samples from two tests. The total TCDF concentrations found in the stack emissions (particulate material and gaseous emissions) ranged from 0.08 to 0.51 ng/dscm and emission factors ranged from 0.28 to 1.88 ng total TCDFs/kg of spent carbon feed. TCDFs were detected in the dryer off-gas cyclone catch from all four tests at concentrations ranging from 0.48 to 3.33 ng total TCDFs/g. Concentrations of 2,3,7,8-TCDF in the effluent samples are reported, however other TCDF isomers could not be specifically identified because reference standards are not available.

One measurement of PCBs in the natural gas used to fire the carbon reactivation unit was performed during the test period. PCBs were detected in the natural gas supply at a concentration of 2.6 micrograms per standard cubic meter (μg/scm). The principal PCBs detected in the natural gas were tri- and tetrachlorobiphenyls.

This report was submitted in fulfillment of Contract No. 68-02-3487, Work Assignment No. 16 by Battelle-Columbus Laboratories under sponsorship of the U.S. Environmental Protection Agency. This report covers the period May 1, 1982 to April 15, 1983, and work was completed as of December 15, 1982.

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LIST OF ABBREVIATIONS

ABBREVIATIONS

kg	-- kilograms
g	-- grams
mg	-- milligrams
μg	-- micrograms
ng	-- nanograms
L	-- liters
mL	-- milliliters
μL	-- microliters
dscf	-- dry, standard cubic feet (20°C, 760mm Hg)
ft/sec	-- feet per second
cfm	-- cubic feet per minute
scf	-- standard cubic feet (20°C, 760mm Hg)
m^3	-- cubic meters
dscm	-- dry, standard cubic meters (20°C, 760mm Hg)
m/sec	-- meters per second
scm	-- standard cubic meters (20°C, 760mm Hg)
ev	-- electron volts
ms	-- milliseconds
cm	-- centimeters
amu	-- atomic mass units
μ	-- micron
ppb	-- parts per billion
ppt	-- parts per trillion

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SECTION 1

INTRODUCTION

Activated carbon has been used for many years to remove taste and odor components from drinking water. More recently, studies have focused on activated carbon treatment to remove potentially hazardous trace organics from municipal water supplies. In this regard, the Cincinnati Waterworks, in conjunction with the U.S. Environmental Protection Agency (EPA) has been conducting a study of water purification with granular activated carbon (GAC). Carbon used for water treatment is thermally reactivated in a fluidized-bed furnace and reused in the water purification process. During carbon reactivation, organic compounds could be released into effluent streams that will enter the environment. A part of the total evaluation of the net gains achieved by removing trace organics from water must include an examination of the potential for causing exposure to the public via alternate pathways. Substances removed from the water must be properly handled regardless of the treatment methodology used. Thus it is important that effluent streams from the reactivation process be characterized to determine whether hazardous compounds are present.

The purpose of this program was to determine quantitatively tetrachlorodibenzo-p-dioxins (TCDDs) and tetrachlorodibenzo furans (TCDFs) in all effluent streams from the Cincinnati Waterworks reactivator unit during a carbon reactivation cycle. The possible presence of TCDDs and TCDFs in the spent carbon feedstock, and PCBs, in the natural gas supply used to fire the reactivator were also evaluated.

The program involved collection of representative effluent stream, feedstock, and natural gas samples from the Cincinnati Waterworks fluidized-bed unit during an approximate 90-hour carbon reactivation cycle. Subsequently, laboratory analyses of the samples were performed by combined high resolution capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS) to determine (1) 2,3,7,8-TCDD and 2,3,7,8-TCDF concentrations, (2) total TCDD and total TCDF concentrations, and (3) the TCDD and TCDF isomeric composition (i.e. the number and, when possible, the identity of isomers resolved in the analyses).

The data generated from this work will be helpful in evaluating any environmental and health hazards associated with effluents from reactivation of carbon used in municipal water treatment. Currently the city of Cincinnati is considering installation of a carbon purification system to treat the total municipal water supply. Thus it is important at this time to determine whether the reactivation process (an economic factor in the overall treatment costs) is environmentally acceptable.

SECTION 2

CONCLUSIONS

The sampling and analytical methods used in the study performed very satisfactorily in the judgement of the investigators and as shown by the quality assurance data. Thus, the data obtained from the study should provide an adequate basis for preliminary evaluation of the environmental impact of the fluidized-bed GAC reactivation system at the Cincinnati Waterworks.

SECTION 3

RECOMMENDATIONS

This work represents a preliminary effort to determine TCDDs and TCDFs in the effluent streams of the Cincinnati Waterworks carbon reactivation system. Similar studies of this process (and other carbon reactivation processes) may be required to answer future questions regarding the overall environmental impact of water purification with granular activated carbon. The following recommendations should be considered in conducting future studies.

1. Tests should be performed to determine TCDF and TCDD emissions during reactivation of GAC which has undergone typical service and storage cycles. Tests should also be performed to evaluate seasonal effects on the composition and levels of the emissions.

2. The spent carbon feed should be analyzed for organic compounds (e.g. trichlorophenols) that may yield TCDDs and/or TCDFs in the reactivation process. From these data, it might be possible to determine if TCDDs and TCDFs in the effluents originate from precursor compounds in the spent carbon.

3. If future plans include using fuel oil as well as natural gas to fire the carbon reactivation system, TCDD and TCDF emission measurements should be made during operation of the unit on fuel oil.

SECTION 4

EXPERIMENTAL PROCEDURES

GENERAL EXPERIMENTAL APPROACH

Four tests were performed to determine TCDD and TCDF emissions from the Cincinnati Waterworks carbon reactivation unit. The tests were conducted during reactivation of a batch of carbon which had been used to adsorb trace organics from Cincinnati municipal drinking water. During each test, samples were acquired from all effluent streams and the spent carbon feedstock. The process streams which were sampled included:

(1) Stack effluent (gases and particulate material) from the fluidized-bed unit after passing through the Venturi and tray scrubbers.

(2) Fine carbon removed from the drying zone off-gas by the cyclone.

(3) Combined liquid discharge from the Venturi and tray scrubbers.

(4) Overflow water from the reactivated carbon quench tank.

(5) Spent carbon feed to the reactivator unit.

A modified Method 5 train was used to sample the stack effluent stream. Grab sampling techniques were used to collect samples of the other effluent streams and the spent carbon feedstock (Items 2-5 above). During one test, the natural gas supply used to fire the fluidized-bed reactivator was sampled with a solid sorbent trap to screen for the presence of polychlorinated biphenyls (PCBs).

The effluent stream and spent carbon feed samples were analyzed by extraction, removal of co-extracted interferences, and determination of TCDDs and TCDFs by combined high resolution gas chromatography/high resolution mass spectrometry. Analyses of the sorbent trap used to sample the natural gas supply were done by extraction and determination of the PCBs by combined high resolution gas chromatography/mass spectrometry.

The following sections provide more detailed descriptions of the carbon reactivation process and the sampling and analytical procedures that were employed in the study.

PROCESS DESCRIPTION AND OPERATING CONDITIONS

A schematic diagram of the Cincinnati Waterworks fluidized-bed carbon reactivation system is presented in Figure 1. The wet spent carbon is fed into the fluidized bed unit by a dewatering screw and first passes through a drying zone which is maintained at about 149°C (300°F). The carbon then passes into a fluidized reactivation zone in which the temperature is closely controlled at about 816°C (1500°F) by either natural gas- or fuel oil-fired burners. During this study, the unit was fired with natural gas.

Fine carbon particles emitted from the dryer off-gas stream are removed by a cyclone. The reactivation/incinerator zone off-gas stream passes through serially-connected Venturi and tray scrubbers and then is emitted to the atmosphere through a stack. The regenerated carbon flows into a water quench tank and is recovered as a slurry for future use.

Reactivator operating data were recorded and archived by Cincinnati Waterworks personnel during the test program. A summary of operating conditions during the test periods is given in Table 1. Stack gas parameters determined by EPA Method 5 procedures are shown in Table 2.

The emission tests were performed during reactivation of about a 18,144 kg (40,000 lb) batch of WestVaco bituminous carbon (12 x 40 mesh) which had been used for treatment of Cincinnati municipal water. The carbon was in service in a contactor bed treatment system for about 1 year. Approximately 6 months elapsed from the time the carbon was taken out of service and the reactivation was performed.

SAMPLING PROCEDURES

Stack Effluent

A modified Method 5 train shown schematically in Figure 2 was used to sample the stack effluent. The train is similar to a system which has been used to assess organic emissions from combustion sources (1). The modification to the conventional Method 5 train consisted of the addition of a water-jacketed condenser and XAD-2 trap between the filter and the first impinger. Cooling water to the condenser and XAD-2 trap jacket was supplied by a submersible pump located in the impinger ice bath. Liquid which condensed and passed through the XAD-2 resin bed was collected in a reservoir and retained for TCDD/TCDF analyses.

Four stack emission tests were performed. During each test, sampling was performed isokinetically at a single point in the stack in a region of average velocity (ΔP) as determined from the preliminary traverse data shown in Appendix A. Sampling was performed according to the EPA Method 5 procedures given in the FEDERAL REGISTER (2). Carbon dioxide (CO_2) and oxygen (O_2) concentrations in the stack gas were performed at least four times during each Method 5 test using Fyrite analyzers. Results of the CO_2 and O_2 measurements and sampling system operating data recorded during each of the

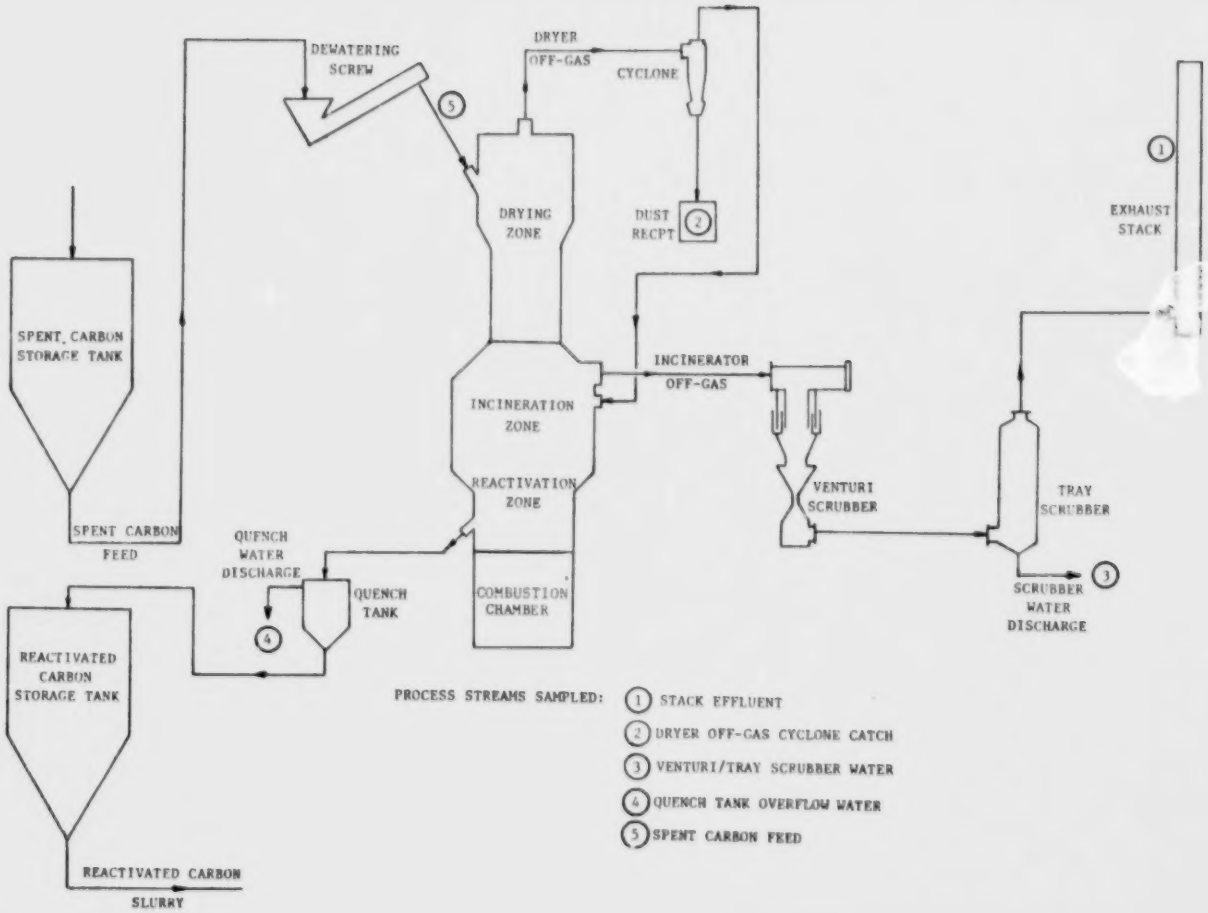

Figure 1. Cincinnati Waterworks carbon reactivation system.

TABLE 1. SUMMARY OF OPERATING CONDITIONS DURING EMISSION TESTS[a]

Test	Spent Carbon Feed[b]		Nat.Gas Feed[c]		Comb. Chamber		Reactivator	Incinerator		Dryer Bed	Scrubber Gas Temp, °F	
No.	Rate, lbs/hr	Total, lbs	Rate, cfm	Total, cf	Temp, °F	O_2, %	Bed Temp., F	Temp, °F	O_2, %	Temp, °F	Venturi Out[d]	Tray Out
1	585	2340	36.4	8736	1730	0.1	1550	1410	2.0	306	189	129
2	585	2340	35.4	8500	1740	0.0	1560	1440	1.9	310	190	131
3	--[e]	--[e]	30.8	7400	1750	0.0	1585	1370	1.7	314	186	119
4	585	2340	34.2	8200	1627	0.0	1590	1490	2.2	289	190	132

(a) Data are totals or averages for the approximate time periods during which the emission tests were performed.

(b) Multiply by 0.4536 to convert lbs or lbs/hr to kg or kg/hr

(c) Multiply by 0.02832 to convert cf or cfm to m^3 or m^3/min.

(d) Venturi inlet thermocouple not operating properly during tests; venturi inlet temperature typically 30°(F) greater than outlet.

(e) Spent carbon feed reduced during this test because of eductor plugging. Feed rate unknown.

TABLE 2. SUMMARY OF STACK GAS PARAMETERS

Parameter	Test No. 1	Test No. 2	Test No. 3	Test No. 4
CO_2 in Stack Gas, % by Vol.	12.76	12.95	11.8	14.03
O_2 in Stack Gas, % by Vol.	4.6	3.5	4.0	3.5
H_2O in Stack Gas, % by Vol.	8.3	8.2	6.7	9.4
Avg. Stack Gas Temp., $^oC(^oF)$	42(108)	43(109)	40(104)	44(112)
Stack Gas Velocity, ft/sec	30.93	31.20	23.39	32.96
Stack Gas Velocity, m/sec	9.43	9.51	7.13	10.05
Volumetric Flow Rate, dscf/hr	34,756	35,037	26,872	36,232
Volumetric Flow Rate, dscm/hr	984	992	761	1,026

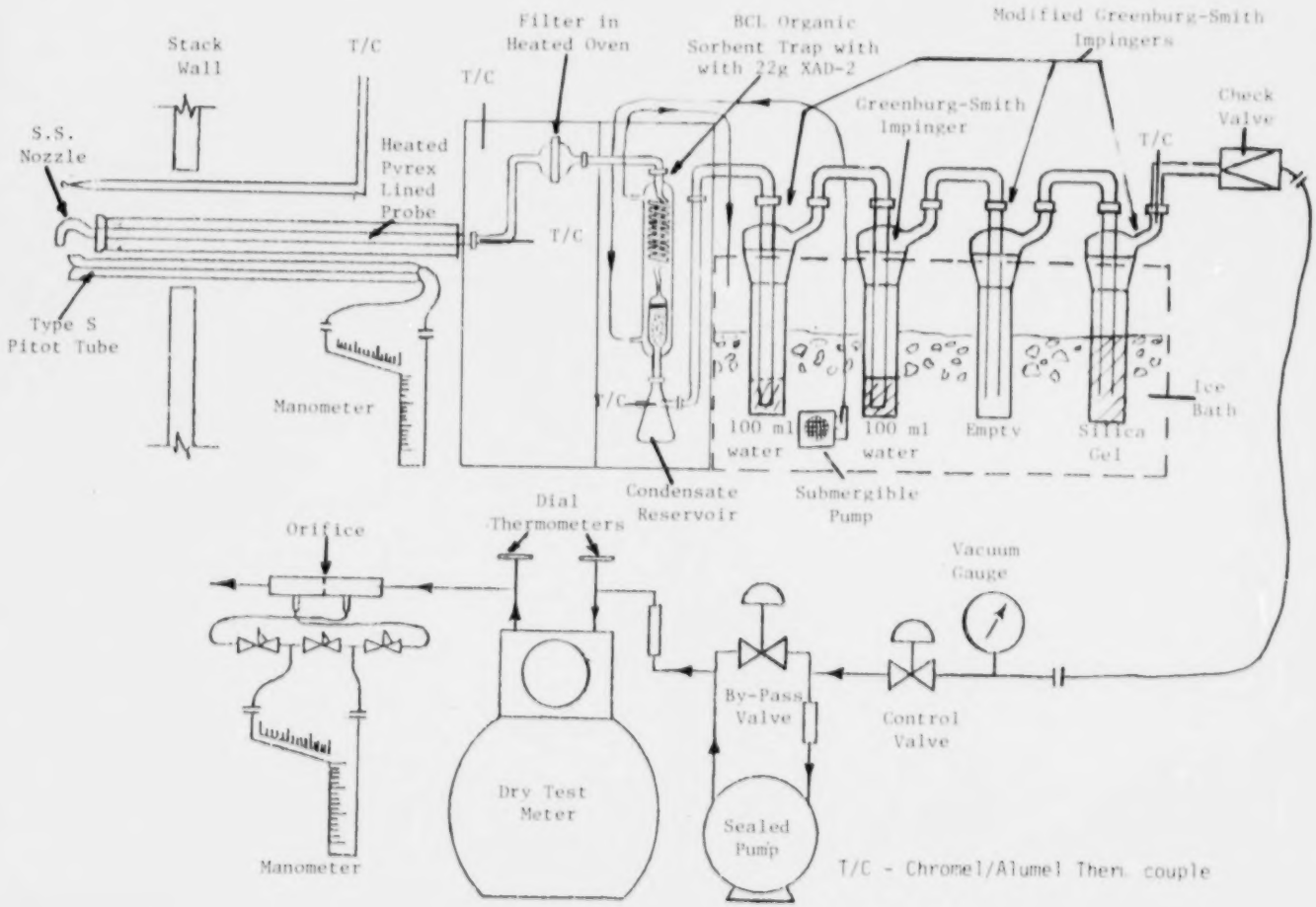

Figure 2. Modified Method 5 sampling train

tests are given in Appendix A. A summary giving test dates and times and the modified Method 5 operating conditions for each test is presented in Table 3.

TABLE 3. SUMMARY OF MODIFIED METHOD 5 SYSTEM OPERATING CONDITIONS

Parameter	Test No. 1	Test No. 2	Test No. 3	Test No. 4
Test Date	8/30/81	8/31/82	8/31/82	9/01/82
Time Started, hr EDT	1323	0847	1443	0915
Time Completed, hr EDT	1723	1247	1843	1315
Sampling Time, min	240	240	240	240
Sample Volume, dscf	177.522	194.606	142.971	176.115
Sample Volume, dscm	5.027	5.511	4.049	4.998
% Isokinetic Sampling Rate	94.6	103.0	98.7	90.1
Avg. Probe Temp., $^{o}C(^{o}F)$	116(241)	118(245)	126(258)	122(252)
Avg. Filter Box Temp., $^{o}C(^{o}F)$	121(249)	121(250)	123(254)	128(262)
Avg. XAD-2 Outlet Temp., $^{o}C(^{o}F)$	7(45)	7(44)	7(44)	8(47)

Following the stack tests, the components of the sampling train were sealed and moved to a clean laboratory area at the Cincinnati Waterworks to conduct sample recovery operations. Recovery of the various samples from the sampling system was performed as shown in Figure 3. Sample recovery data are presented in Appendix A.

Cyclone Catch and Spent Carbon Feedstock

Approximately 75-g grab samples of the cyclone catch and 130-g grab samples of the spent carbon feedstock were taken at 30-minute intervals during each modified Method 5 test. The eight grab samples taken during each test were combined and homogenized by thorough mixing with a stainless steel spatula to provide a single composite sample of the respective process streams. The cyclone catch sample was taken from the receptacle used to collect carbon fines removed by the cyclone. The receptacle was emptied after taking each grab sample. Thus, each grab sample represented the cyclone catch collected over a 30-minute interval. The spent carbon feed sample was acquired at a port in the drop line from the dewatering screw to the dryer. Field recovery procedure for the cyclone catch and spent carbon feed samples is shown in Figure 3. Cyclone catch and spent carbon feed sampling data are given in Appendix A.

Scrubber and Quench Water

Sixteen-ounce grab samples of the scrubber and quench water samples were taken at 30-minute intervals during each modified Method 5 test. The eight grab samples taken during the test were combined and homogenized by shaking to yield a single composite sample of the respective effluent streams as shown in Figure 3.

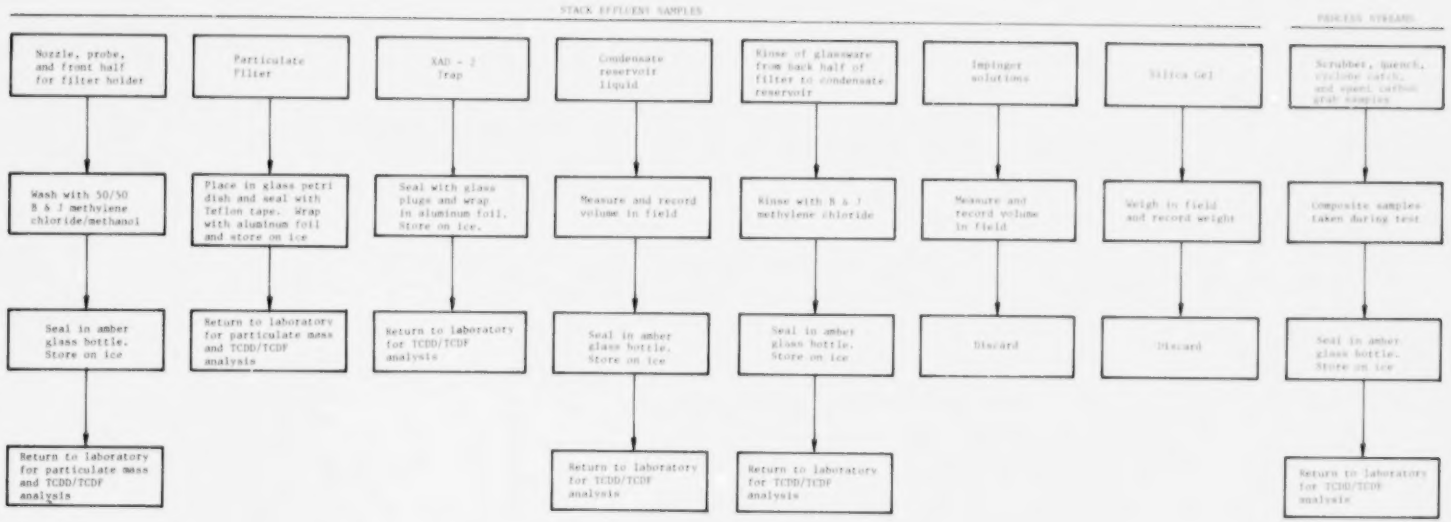

Figure 3. Field sample recovery procedures

The grab samples were obtained from taps in the scrubber and quench water effluent lines. The tap lines were thoroughly flushed with the scrubber or quench water just prior to sample collection. Sample collection bottles were rinsed three times with the scrubber or quench water prior to acquisition of a sample. Scrubber and quench water sampling data are presented in Appendix A.

Natural Gas Supply

During Method 5, Test No. 3, the natural gas supply used to fire the fluidized-bed reactivation system was sampled to determine if polychlorinated biphenyls (PCBs) were present. A sample stream taken from the supply line to the boiler was passed through a 22-gram XAD-2 trap operated at ambient temperature. Sample volume was measured with a calibrated dry gas meter downstream from the XAD-2 trap. Sampling was performed for a 2-hour period during the 4-hour modified Method 5 Test. Sampling was performed at a flow rate of about 0.012 m^3/min (0.43 cfm); total sample volume was 1.4 scm (49.469 scf). The PCB sampling data are given in Appendix A.

Preparation of Sampling Equipment and Sample Containers

All glassware used for sampling and sample containment was cleaned prior to use by the following steps:

(1) Wash in a hot synthetic detergent solution

(2) Rinse with hot tap water

(3) Soak for at least 30 minutes in hot 40-50°C (104-122°F) chromic acid solution*

(4) Rinse with hot tap water

(5) Rinse with distilled water

(6) Rinse with methanol (Burdick & Jackson "Distilled in Glass")

(7) Rinse with methylene chloride (Burdick & Jackson "Distilled in Glass")

(8) Dry with a stream of purified nitrogen.

After drying, all equipment and containers were sealed to prevent contamination prior to sampling.

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* 15% HNO_3 was used in this step to clean the modified Method 5 stainless steel sampling nozzle.

Sample Containment and Preservation

Following collection, all samples were transferred to amber glass bottles with Teflon cap liners or clear glass containers wrapped with aluminum foil. The samples were then placed on ice (~0°C or ~32°F) in ice chests for storage during the sample collection period and during shipment to Battelle's laboratories. Upon arrival at the laboratory, all samples were transferred from the ice chests to refrigerated storage where they were held until start of the analysis. Analysis of the samples commenced 6 days after the samples were delivered to the laboratory.

ANALYTICAL PROCEDURES

TCDD and TCDF Analyses

Overview of Sample Analysis Steps--

TCDD and TCDF analyses of the samples involved three basic steps: (1) extraction of the TCDDs and TCDFs from the sample matrix; (2) sample clean-up to remove co-extracted interferences; and (3) analysis of TCDDs and TCDFs by HRGC/HRMS. Several intermediate steps were required to reduce sample volume and to exchange the extract to a solvent suitable for subsequent operations. Figures 4 and 5 show the sequence of the steps in the TCDD/TCDF analysis procedure. Figure 4 shows the sample preparation/ combination and extraction steps for each sample type. The cleanup and analysis steps which were common to all sample types are shown in Figure 5.

Extraction Procedures--

Two extraction techniques were used for the sample analyses. The XAD-2 resin/organic module rinse samples and the particulate samples were Soxhlet-extracted with methylene chloride and benzene, respectively, while liquid-liquid extraction was used for the scrubber and quench water samples. Methylene chloride is commonly used as the solvent for most XAD-2 Soxhlet extractions, and has been shown to be an efficient solvent for the extraction of TCDD/TCDF. The particulate samples, however due to stronger adsorption require an aromatic solvent such as benzene to assure efficient recovery of the TCDDs and TCDFs (3). Prior to extraction, each sample was spiked with 2,3,7,8-TCDD-$^{13}C_{12}$ to serve as an internal standard for recovery calculations and quantification. A description of the extraction methodologies follows.

The particulate samples included the combined modified Method 5 filter and rinse residue, the dryer off-gas cyclone catch, and spent carbon feed samples. Initially, 5-gram aliquots of the cyclone catch and spent carbon feed were analyzed, but recoveries determined from the internal standard were very low. The following modifications were made in the extraction procedure for subsequent analyses of these samples and acceptable recoveries (>50%) were obtained.

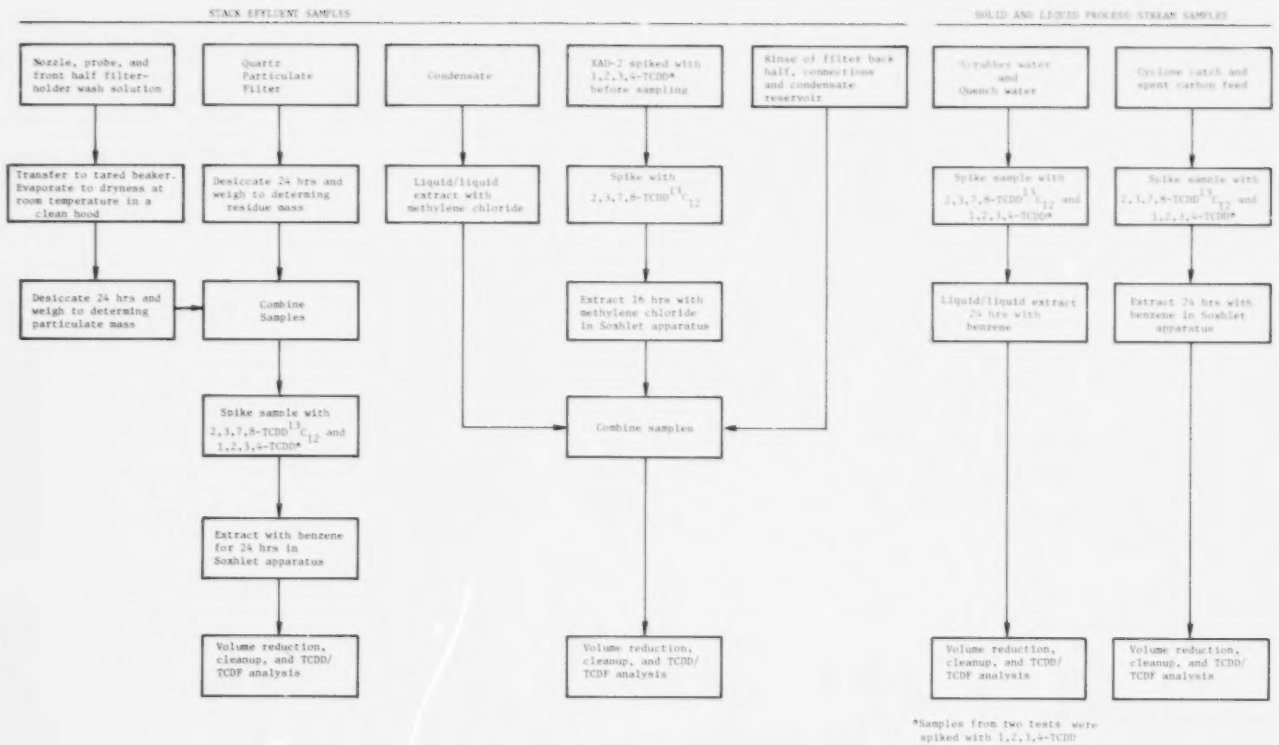

Figure 4. Sample combination/preparation steps

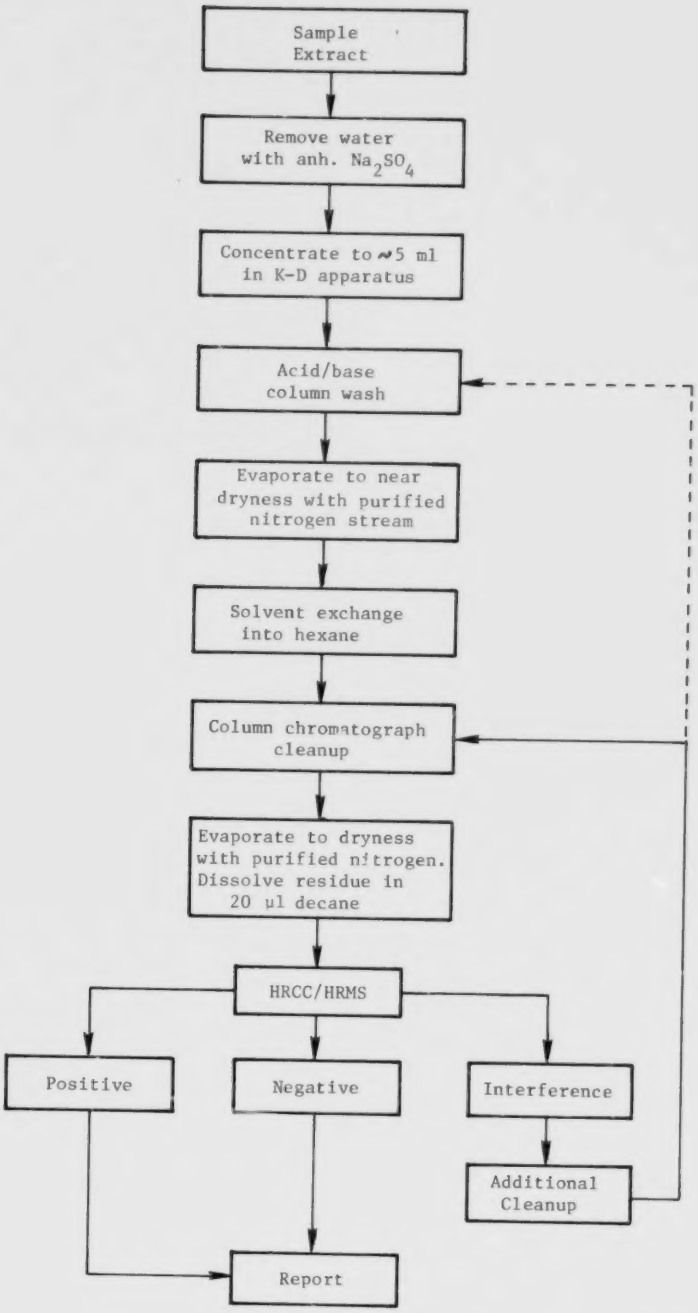

Figure 5. Sample clean-up and analysis steps

1) The quantity of 2,3,7,8-TCDD-$^{13}C_{12}$ internal standard added prior to extraction was increased from 2 ng to 20 ng.

2) Sample size was decreased from 5 g to 2 g.

3) Fritted glass thimbles instead of cellulose were used to achieve better solvent-sample contact.

4) Extraction time was increased from 16 hrs. to at least 24 hrs.

For the extraction, the particulate samples were transferred into the extraction thimble and placed into the Soxhlet apparatus where enough benzene was added to cover the samples. The 2,3,7,8-TCDD-$^{13}C_{12}$ internal standard was added and the samples were extracted with benzene. Teflon tape was used to seal the ground glass joints to minimize evaporative losses. Following the extraction, the benzene was transferred to an Erlenmeyer flask using three 15 ml rinses of fresh benzene. The benzene was dried by slowly adding approximately 1 g of anhydrous sodium sulfate. The sodium sulfate was added slowly to prevent the formation of clumps which can entrap the TCDDs and TCDFs. The dried extract was then transferred to a Kuderna-Danish (K-D) apparatus using three 15 ml rinses of fresh benzene and concentrated to approximately 5 ml volume. The extract was stored in a glass stoppered bottle at $0^{o}C(32^{o}F)$ until processed through the acid/base and chromatographic clean-up steps.

The XAD-2 resin samples were Soxhlet-extracted using methylene chloride. The resin was transferred into a pre-extracted cellulose extraction thimble, spiked with 2 ng of 2,3,7,8-TCDD-$^{13}C_{12}$ and allowed to extract for at least 16 hours. The methylene chloride extract was dried with anhydrous sodium sulfate and concentrated in a K-D apparatus to a volume of approximately 5 ml following the same procedure as described for the particulate extracts.

The scrubber water and quench water samples were extracted by liquid-liquid extraction using benzene. A one-liter aliquot of each sample was transferred to a two-liter glass bottle and spiked with 20 ng of 2,3,7,8-TCDD-$^{13}C_{12}$. A Teflon-coated magnetic stirring bar was added and the sample was stirred for about 30 minutes. A 300 ml portion of benzene was added and the stirring resumed for at least 24 hours. The benzene extract was then transferred to an Erlenmeyer flask using three 50 ml rinses of fresh benzene, dried with anhydrous sodium sulfate, and concentrated to 5 ml using the procedure described for the particulate extracts. Low recoveries (possibly due to solids in the samples) were obtained in initial analyses using 2 ng of internal standard and extraction times of approximately 16-18 hrs. Acceptable recoveries (>50%) were achieved using the extraction procedure described above.

Sample Clean-Up Procedures--

The benzene extracts of the modified Method 5 particulate, scrubber water, quench water and spent carbon feed samples and the methylene chloride extracts of the condensate and XAD-2 resin (including the methylene chloride organic module rinse) were treated to remove interferences using a series of liquid chromatography (LC) columns. The first column used to treat the

extracts was a multilayered silica column which served to remove acidic, basic, and easily oxidized compounds. The column contained approximately 4 g of silica treated with sulfuric acid, 4 g of silica treated with sodium hydroxide, and 2 g of activated silica. The column was packed dry and pre-rinsed with 25 ml of hexane prior to use.

The sample extracts were transferred onto the silica column using three 5 ml rinses of hexane/benzene (50:50). The extract was eluted with 50 ml of hexane/benzene (50:50) and the total eluate (about 70 ml) was collected. The eluate was then concentrated nearly to dryness under a gentle stream of purified nitrogen and dissolved in about 5 ml of hexane.

The extract (in hexane) was transferred to a 5 g activated alumina clean-up column. The column was pre-rinsed with 25 ml hexane followed by the addition of the sample. The column was sequentially eluted with 25 ml portions of hexane, hexane/carbon tetrachloride (1:1), and hexane/methylene chloride (1:1). The hexane/methylene chloride fraction was collected and stored at 10°C (50°F) until analyzed. Just prior to analysis, the hexane/methylene chloride solution was evaporated to dryness at ambient temperature using a stream of purified nitrogen. The residue was then dissolved in 20 µl of n-decane (accurately measured). Analysis for TCDD and TCDF was performed on a 2 µl aliquot of the n-decane solution.

HRGC/HRMS Analysis of TCCDs and TCDFs--

The extracts were analyzed for TCDDs and TDCFs using combined capillary column gas chromatography/high resolution mass spectrometry (HRGC/HRMS). A Carlo Erba Model 4160 gas chromatograph interfaced to a VG Model 7070H high resolution mass spectrometer was used for the analyses. Typical operating parameters were:

Mass Spectrometer		
Electron Impact Ionization	-	70 ev
Mass Resolution	-	10,000-12,000 ($m/\Delta m$, 10% valley)
Source Temperature	-	200°C (392°F)
Multiple Ion Detection Mode	-	25 ms dwell time, total cycle time 200 ms
Gas Chromatograph		
Column	-	30 meter DB5 or 60 meter SP2340 Fused Silica Column
Carrier Flow	-	30 cm/sec (helium)
Injector Temperature	-	275°C (527°F)
Temperature Program	-	140°C (284°F) for 3 minutes; linear increase to 275°C (527°F) at 20°C (36°F)/ minute and hold at 275°C (527°F) for 10 minutes
Transfer Line	-	290°C (554°F)

A VG Model 2035 Data System was used to control the multiple ion detection (MID) acquisition and to acquire the time-intensity data. Seven ion masses shown below were monitored.

318.9793	Reference Lock Mass (Perfluorokerosene)
303.9016	TCDF
305.8987	TCDF
319.8965	TCDD
321.8936	TCDD
331.9368	2,3,7,8-TCDD-$^{13}C_{12}$ internal standard
333.9338	2,3,7,8-TCDD-$^{13}C_{12}$ internal standard

The criteria for a positive identification were:

1. Correct chromatographic retention time within $\pm$ 2 seconds based on 2,3,7,8-TCDD-$^{13}C_{12}$ internal standard and external standards (1,3,6,8-TCDD, 1,3,7,9-TCDD, 2,3,7,8-TCDD, or 2,3,7,8-TCDF)

2. Response at both m/z 304 and m/z 306 for TCDFs

3. Response at both m/z 320 and m/z 322 for TCDDs

4. Isotopic ratio correct for a tetrachloro compound (0.77 $\pm$ 10%)

5. Signal-to-noise ratio equal to or greater than 2.5 to 1.0.

The specific TCDD isomers, 1,3,6,8-TCDD, 1,3,7,9-TCDD, and 2,3,7,8-TCDD were identified by criteria 1, 3, 4, and 5. The 2,3,7,8-TCDF was identified by criteria 1, 2, 4, and 5. Other TCDD/TCDF isomers were identified using criteria 2, 3, 4, and 5 listed above and the requirement that retention times were within windows determined from previous analyses of mixtures of TCDD/TCDF isomers.

The TCDDs and TCDFs were quantified by comparison of the responses (selected ion current profile peak areas), of the native TCDD/TCDF isomers with the response of the 2,3,7,8-TCDD-$^{13}C_{12}$ internal standard. Thus all reported TCDD/TCDF levels were corrected for the recovery efficiencies. A response factor of 1.0 relative to the internal standard was assumed for all TCDD and TCDF isomers.

PCB Analysis--

Analysis for PCBs collected from the natural gas supply line by trapping on XAD-2 was performed by solvent extraction of the solid sorbent and determination of PCBs in the extract by combined high resolution gas chromatography/mass spectrometry (HRGC/MS). The XAD-2 was extracted with methylene chloride for about 16 hours in a Soxhlet apparatus. The extract was then reduced in volume to 0.2 mL in a Kuderna-Danish apparatus followed by evaporation with a stream of purified nitrogen. The extract was next spiked with 2 μg of dibromobiphenyl (internal standard) and analyzed by HRGC/MS using a Finnigan Model 3200 system with an INCOS data system. The HRGC/MS operating conditions were as follows:

Mass Spectrometer

Electron Impact Ionization - 70 ev
Source Temperature - 100°C (212°F)
Scan Range - 40 to 540 amu
Scan Rate - 0.75 sec/scan
Mode - Full spectrum scan

Gas Chromatograph

Column - 30 meter, DB-5 Fused Silica
0.248 in. ID, 0.25μ film (J&W Scientific)
Carrier flow - 30 cm/sec
Temperature program - 45°C (113°F) for 4.5 minutes; linear increase to 300°C (572°F) at 8°C (14°F)/minute
Injector temperature - 300°C (572°F)
Transfer Line Temperature - 300°C (572°F)
Injector - Grob-type, splitless
Injection volume - 2 μl

The PCB concentration in the XAD-2 extract was determined by comparison to a standard solution comprised of known quantitites of the PCB isomers listed in Table 4. The response factor for each level of chlorination was determined by summation of the ion currents for the two characteristic masses specific to that chlorination level (see Table 4). The response factors obtained from the standard solution were applied to the sample extract to determine the concentration of all isomers at each level of chlorination. The response for all isomers of a given chlorination level in the sample was assumed to be the same as the response for the isomer used as the reference standard.

Each selected ion current profile for a given level of chlorination was manually examined to assure that both quantitation masses were present and that they maximized at the same scan number. The isotope ratio for the two masses was also considered.

TABLE 4. COMPOSITION OF PCB STANDARD[a]

Level of Chlorination	Isomer	Masses used for Quantitation (m/z)
1	2-chlorobiphenyl	188,190
2	2,6-dichlorobiphenyl	222,224
3	2,2'5-trichlorobiphenyl	256,258
4	2,2'5,6'-tetrachlorobiphenyl	290,292
5	2,2',3,5,6-pentachlorobiphenyl	326,328
6	2,2',4,4',5,6-hexachlorobiphenyl	360,362
7	2,2',3,4,4',5,6-heptachlorobiphenyl	394,396
8	2,2'3,3'4,5,6,6'-octachlorobiphenyl	428,430
Internal Standard	4,4'-dibromobiphenyl	310,312

(a) Standard solution prepared from individual PCB isomers obtained from Ultra Scientific.

SECTION 5

RESULTS AND DISCUSSION

STACK PARTICULATE EMISSIONS

The results of the particulate mass emission measurements obtained from the modified Method 5 stack tests are given in Table 5.

TABLE 5. PARTICULATE MASS EMISSION DATA

	Test 1	Test 2	Test 3	Test 4
Filter Catch Wt., mg	5.0	7.2	6.5	9.6
Rinse Residue Wt., mg	15.7	12.5	0.0	8.4
Total Particulate Wt., mg	20.7	19.7	6.5	18.0
Particulate Mass Conc. in Stack Gas, mg/dscm (a)	4.12	3.58	1.61	3.61
Particulate Emission Rate, g/hr (b)	4.05	3.55	1.22	3.70
Emission Factor, g/kg Spent Carbon (b)	0.015	0.013	(c)	0.014

a) Calculated from sample volumes given in Table 3.
b) Calculated from reactivator operating data given in Tables 1 and 2.
c) Spent carbon feed rate unknown. Eductor line plugging occurred during test.

TCDD AND TCDF MEASUREMENTS

The results of the measurements of TCDDs and TCDFs in the reactivator effluent stream samples and the spent carbon feed are shown in Tables 6 and 7. Results are reported for 2,3,7,8-TCDD and total TCDD, and 2,3,7,8-TCDF and total TCDF concentrations measured in the samples. The number of TCDD or TCDF isomers identified in each sample is shown in parentheses beside

TABLE 6. TCDD AND TCDF STACK EMISSION DATA[a]

	Stack Particulate Emissions				Stack Gas Emissions				Total Stack Emissions			
	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 4	Test 1	Test 2	Test 3	Test 4
Concentration in Stack Gas, ng/dscm												
2,3,7,8-TCDD	0.21	0.03	0.01	0.13	<0.01	0.01	<0.01	<0.01	0.21	0.04	0.01	0.13
Total TCDD	0.21(1)	0.24(6)	0.06(7)	0.13(2)	<0.02	0.06(4)	<0.02	<0.02	0.21	0.30	0.06	0.13
2,3,7,8-TCDF	0.24	<0.01	<0.01	0.06	<0.02	<0.02	<0.01	<0.01	0.24	<0.01	<0.01	0.06
Total TCDF	0.38(3)	<0.02	0.40(4)	0.26(4)	0.13(6)	0.08(11)	<0.02	<0.02	0.51	0.08	0.40	0.26
Concentration in Stack Particulates, ng/g												
2,3,7,8-TCDD	51.2	8.4	4.3	35.5	--	--	--	--	51.2	8.4	4.3	35.5
Total TCDD	51.2	65.9	37.3	35.5	--	--	--	--	51.2	65.9	37.3	35.5
2,3,7,8-TCDF	58.0	<2	<7	17.2	--	--	--	--	58.0	<2	<7	17.2
Total TCDF	92.2	<4	245.3	72.3	--	--	--	--	92.2	<4	245.3	72.3
Emission Rates, ng/hr												
2,3,7,8-TCDD	207	30	5	131	<8	6	<4	<10	207	36	5	131
Total TCDD	207	234	46	131	<16	60	<8	<20	207	294	46	131
2,3,7,8-TCDF	235	<7	<9	64	<20	<16	<2	<2	235	<16	<9	64
Total TCDF	373	<14	299	268	126	75	<3	<4	499	75	299	268
Emission Factors, ng/kg Spent Carbon												
2,3,7,8-TCDD	0.77	0.11	(b)	0.49	<0.03	0.02	(b)	<0.04	0.77	0.13	(b)	0.49
Total TCDD	0.77	0.88	(b)	0.49	<0.06	0.23	(b)	<0.08	0.77	1.11	(b)	0.49
2,3,7,8-TCDF	0.89	<0.03	(b)	0.24	<0.08	<0.06	(b)	<0.01	0.89	<0.06	(b)	0.24
Total TCDF	1.41	<0.05	(b)	1.01	0.47	0.28	(b)	<0.02	1.88	0.28	(b)	1.01

(a) Number of TCDD or TCDF isomers detected in samples are indicated in parentheses.

Less than (<) values reported indicate that TCDDs or TCDFs were not detected; the values shown are estimated minimum detectable concentrations (MDC). MDC's vary with sample type and interferences in samples.

(b) Spent carbon feed rate unknown. Eductor line plugging occurred during test.

TABLE 7. TCDD AND TCDF CONCENTRATIONS IN CARBON REACTIVATOR SAMPLES[a]

Test No.	Analyte	Cyclone Catch, ng/gram	Scrubber Water, ng/liter	Quench Water, ng/liter	Spent Carbon Feed, ng/gram
1	2,3,7,8-TCDD	<0.01	<0.02	<0.05	<0.05
	Total TCDD	3.30(1)	<0.04	<0.1	<0.1
	2,3,7,8-TCDF	<0.02	<0.05	<0.03	<0.02
	Total TCDF	0.48(7)	<0.1	<0.06	<0.04
2	2,3,7,8-TCDD	<0.04	0.07	<0.02	<0.02
	Total TCDD	0.20(1)	0.21(3)	<0.04	<0.04
	2,3,7,8-TCDF	2.71	<0.01	<0.06	<0.04
	Total TCDF	3.33(10)	<0.02	<0.12	<0.08
3	2,3,7,8-TCDD	0.01	0.06	<0.02	<0.02
	Total TCDD	4.14(6)	0.12(5)	<0.04	<0.04
	2,3,7,8-TCDF	1.71	<0.01	<0.03	<0.01
	Total TCDF	2.14(10)	<0.02	<0.06	<0.02
4	2,3,7,8-TCDD	<0.01	<0.02	<0.02	<0.01
	Total TCDD	1.30(1)	<0.04	<0.04	<0.02
	2,3,7,8-TCDF	0.61	<0.05	<0.06	<0.03
	Total TCDF	0.78(4)	<0.1	<0.12	<0.06

(a) Number of TCDD or TCDF isomers detected in samples are indicated in parentheses.

Less than (<) values reported indicate that TCDDs or TCDFs were not detected; the values shown are estimated minimum detectable concentrations (MDC). MDC's vary with sample type and interferences in samples.

the total TCDD or total TCDF concentration. Less than (<) values reported in the tables indicate that TCDDs or TCDFs were not detected in the sample; the value shown is the minimum detection limit. All results in the tables have been corrected to 100% recovery using the internal standard data. In Table 6, concentration, emission rate, and emission factor data are reported for 1) TCDDs and TCDFs in the stack particulates, 2) TCDDs and TCDFs in the stack gas, and 3) TCDDs and TCDFs in the total stack emissions (sum of particulate and gaseous emissions).

TCDD and TCDF concentrations found in the dryer off-gas cyclone catch, scrubber and quench waters, and the spent carbon feed samples are shown in Table 7. TCDD and TCDF analysis of each sample type from Tests 2 and 3 was performed in duplicate. Results of the duplicate analyses are reported in Section 6. The results for these tests given in Table 7 are for single determinations in which the highest recoveries (and presumably more accurate data) were obtained.

Effluent streams in which TCDDs were detected were the stack emissions, the dryer off-gas cyclone catch, and the scrubber water. TCDDs were not detected in any quench water or spent carbon feed samples from the four tests. TCDDs were detected in the stack particulate samples from all four tests and in the vapor phase (XAD-2) sample from one test. The total TCDD concentrations found in the stack emissions (particulate material and gaseous emissions) ranged from 0.06 to 0.3 ng/dscm and emission factors ranged from 0.49 to 1.11 ng total TCDDs/kg of spent carbon feed. TCDDs were detected in the dryer off-gas cyclone catch samples from all four tests and in the scrubber liquid samples from two of the four tests. The total TCDD concentrations found in the cyclone catch ranged from 0.20 to 4.14 ng/g. TCDDs at concentrations of 0.12 and 0.21 ng/L were found in the scrubber water from two tests.

Effluent streams in which TCDFs were detected were the stack emissions and the dryer off-gas cyclone catch. TCDFs were not detected in any scrubber water, quench water, or spent carbon feed samples from the four tests. TCDFs were detected in stack particulate samples from three tests and in the vapor phase (XAD-2) samples from two tests. The total TCDF concentrations found in the stack emissions (particulate material and gaseous emissions) ranged from 0.08 to 0.51 ng/dscm and emission factors ranged from 0.28 to 1.88 ng total TCDFs/kg of spent carbon feed. TCDFs were detected in the dryer off-gas cyclone catch from all four tests at concentrations ranging from 0.48 to 3.33 ng total TCDFs/g.

Summaries of TCDD and TCDF levels (in ppb or ppt) found in the reactivator effluent streams and the spent carbon feed are given in Tables 8 and 9, respectively.

Typical HRGC/HRMS selected ion current profiles of the various effluent stream samples and spent carbon feed are presented in Appendix B. The principal isomers found in the samples were: 1,3,6,8-TCDD, 1,3,7,8-TCDD, 1,3,7,9-TCDD, and 2,3,7,8-TCDD. Some samples contained other unidentified TCDD isomers present at very low levels relative to the isomers listed above.

TABLE 8. SUMMARY OF TCDD CONCENTRATIONS IN CINCINNATI WATERWORKS REACTIVATOR SAMPLES

Regenerator Stream (TCDD Conc. Units)	Test 1		Test 2		Test 3		Test 4	
	2,3,7,8-TCDD	Total TCDD	2,3,7,8-TCDD	Total TCDD	2,3,7,8-TCDD	Total TCDD	2,3,7,8-TCDD	Total TCDD
Spent Carbon Feed (ppb by wt)	ND[a]	ND	ND	ND	ND	ND	ND	ND
Cyclone Catch (ppb by wt)	ND	3.3	ND	0.2	0.01	4.1	ND	1.3
Scrubber Water (ppt by wt)	ND	ND	0.07	0.2	0.06	0.1	ND	ND
Quench Water (ppt by wt)	ND	ND	ND	ND	ND	ND	ND	ND
Stack Emissions								
Particulates (ppt by wt)	0.2	0.2	0.02	0.2	0.01	0.05	0.1	0.1
(ppt by vol)	0.02	0.02	0.002	0.02	0.001	0.004	0.01	0.01
Gas Phase (ppt by wt)	ND	ND	0.01	0.05	ND	ND	ND	ND
(ppt by vol)	ND	ND	0.001	0.004	ND	ND	ND	ND

(a) ND - TCDDs were not detected.

TABLE 9. SUMMARY OF TCDF CONCENTRATIONS IN CINCINNATI WATERWORKS REACTIVATOR SAMPLES

Regenerator Stream (TCDF Conc. Units)	Test 1		Test 2		Test 3		Test 4	
	2,3,7,8-TCDF	Total TCDF	2,3,7,8-TCDF	Total TCDF	2,3,7,8-TCDF	Total TCDF	2,3,7,8-TCDF	Total TCDF
Spent Carbon Feed (ppb by wt)	ND[a]	ND	ND	ND	ND	ND	ND	ND
Cyclone Catch (ppb by wt)	ND	0.5	2.7	3.3	1.7	2.1	0.6	0.8
Scrubber Water (ppt by wt)	ND	ND	ND	ND	ND	ND	ND	ND
Quench Water (ppt by wt)	ND	ND	ND	ND	ND	ND	ND	ND
Stack Emissions								
Particulates (ppt by wt)	0.2	0.3	ND	ND	ND	0.3	0.05	0.2
(ppt by vol)	0.02	0.03	ND	ND	ND	0.03	0.004	0.02
Gas Phase (ppt by wt)	ND	0.1	ND	0.07	ND	ND	ND	ND
(ppt by vol)	ND	0.01	ND	0.006	ND	ND	ND	ND

(a) ND - TCDFs were not detected.

When present, 2,3,7,8-TCDF was identified, however other TCDF isomers could not be specifically identified due to lack of suitable reference standards. In general, there does not appear to be any consistent relationship between the isomeric composition of either the TCDDs or the TCDFs in the various samples.

PCBs IN THE NATURAL GAS SUPPLY

Sampling for PCBs in the natural gas supply used to fire the reactivator unit was performed during modified Method 5 Test No 3. Results of analysis of the XAD-2 trap used to collect the PCBs are shown in Table 10. From these data, the concentration of PCBs in the natural gas supply was 2.6 μg/scm. Mass profiles of the XAD-2 extract and the standards used for the analysis are presented in Appendix B. Tri- and tetrachlorobiphenyls were the principal PCBs detected in the natural gas supply.

TOTAL ORGANIC HALOGENS MEASUREMENTS

Composite reactivated carbon and spent carbon samples obtained during the test program and a sample of virgin carbon of the type being reactivated were analyzed for total organic halogens (TOX) by Ron Dressman, EPA/Cincinnati. Results of the TOX analyses are reported below:

Virgin carbon - not detected
Reactivated carbon - 55 mg/kg
Spent carbon - 657 mg/kg.

As shown by this data, the carbon was heavily loaded with organic halogens with most of these organics removed during reactivation. Only a small fractional amount of TCDD and TCDF were formed from the total TOX adsorbed on the carbon.

TABLE 10. PCB ANALYSIS OF NATURAL GAS SUPPLY

Level of Chlorination	Quantity of PCBs Found, μg	
	Blank (XAD-2)	Natural Gas Sample
Cl_1	<0.03	<0.03
Cl_2	<0.03	0.29
Cl_3	<0.03	1.40
Cl_4	<0.03	1.80
Cl_5	<0.03	0.16
Cl_6	<0.03	<0.03
Cl_7	<0.03	<0.03
Cl_8	<0.03	<0.03
Total	<0.03	3.65

Total sample volume - 1.4 scm (49.47 scf)

PCB concentration in Natural Gas Supply - 2.6 μg/scm (0.07 μg/scf)

SECTION 6

QUALITY ASSURANCE DATA SUMMARY

During the study, a series of quality assurance (QA) activities were conducted according to a Quality Assurance Project Plan (4). The following sections present a discussion and results of these QA activities.

INSTRUMENT CALIBRATIONS

Modified Method 5 Sampling System

Components of the modified Method 5 train used in the sampling program were calibrated according to the procedures described in the FEDERAL REGISTER (2). The calibration data are presented in Appendix C.

HRGC/HRMS System

The HRGC/HRMS system was calibrated by comparing responses of known quantities of 2,3,7,8-TCDD, one of the analytes of interest, with a known quantity of 2,3,7,8-TCDD-$^{13}C_{12}$, the internal standard. Primary calibration of the HRGC/HRMS system were performed with mixtures containing 0.19 ng 2,3,7,8-TCDD-$^{13}C_{12}$ and 2,3,7,8-TCDD at seven levels over the concentration range of .001 to 1.0 ng. Calibration of the HRGC/HRMS system was checked with a combined 2,3,7,8-TCDD/2,3,7,8-TCDD-$^{13}C_{12}$ standard before and after analysis of each batch of five samples. Typical HRGC/HRMS selected ion current profiles of the 2,3,7,8-TCDD/2,3,7,8-TCDD-$^{13}C_{12}$ standard analyzed with the samples are shown in Appendix B.

All solutions of the 2,3,7,8-TCDD standard were referenced to the same gravimetrically-prepared stock solution. The internal standard material, 2,3,7,8-TCDD$^{13}C_{12}$, however, was obtained in solution. Its concentration has been checked with gravimetrically prepared standards of native 2,3,7,8-TCDD from four sources; EPA (R. Harless, HERL/RTP), University of Nebraska, McMaster University, and Monsanto.

The HRMS was tuned to a reference peak from perfluorokerosene (PFK) which was continuously monitored and 2,3,7,8-TCDD peaks were referenced to the PFK peak.

The instrumental response ratio between each TCDD and TCDF isomer observed in the samples and the 2,3,7,8-TCDD-$^{13}C_{12}$ internal standard was measured and the response [factor (1.0 assumed) for each isomer] relative to the internal standard was used to calculate concentration of each TCDD and TCDF isomer.

EXTRACTION EFFICIENCIES

Extraction efficiencies (or recoveries) for the various carbon reactivator samples analyzed during the study are given in Table 11. The extraction efficiencies were calculated from the recovery of the 2,3,7,8-TCDD-$^{13}C_{12}$ internal standard added to each sample before extraction and HRGC/HRMS analysis. As discussed previously, low extraction efficiencies (values in parentheses) were obtained in the initial analysis of cyclone catch, scrubber water, quench water, and spent carbon feed samples. As shown in Table 11, modifications in the extraction procedures described in Section 4 improved the extraction efficiencies for the subsequent samples.

DUPLICATE ANALYSIS

Results of duplicate analyses of the dryer off-gas cyclone catch, scrubber and quench water, and spent carbon feed samples from Tests 2 and 3 are shown in Table 12. Extraction efficiencies are shown in parentheses beside the analytical results. The data are presumed to be more accurate for the determinations in which the higher extraction efficiencies were obtained.

BLANK ANALYSIS

A set of field and laboratory blanks were analyzed along with the carbon reactivator samples. The results of the analysis of the blank samples are presented in Table 13. The Method 5 filter blank was taken from the batch of filters carried to the field site to perform the stack sampling. The methanol/methylene chloride blank combined with the filter blank for analysis was taken from the same batch of reagents and the same Teflon wash bottle used to rinse the components of the Method 5 train. The XAD-2 blank was taken from a set of traps taken to the site to perform the sampling program. The methylene chloride blank combined with the XAD-2 blank for analysis was taken from the same batch of reagent and the same Teflon wash bottle used to rinse the components of the Method 5 vapor phase organic collection module. The benzene and methylene chloride laboratory blanks were taken from the same lot of reagent as that used for extraction of the reactivator samples.

TCDDs and TCDFs were not detected in any of the field or laboratory blanks. Less than (<) values reported in Table 13 are minimum detection levels for TCDDs and TCDFs in the various reagent and material blanks. Minimum detection levels are higher in the filter and XAD-2 blanks due to interferences present in these materials and are lower in relatively pure solvents, benzene and methylene chloride, which were used for the sample extractions.

TABLE 11. EXTRACTION EFFICIENCIES FOR CARBON REACTIVATOR SAMPLES

Test No.	Extraction Efficiency (Recovery), percent [a]					
	Mod Method 5 Filter/Rinse	XAD-2, rinse and Condensate	Cyclone Catch	Scrubber Water	Quench Water	Spent Carbon Feed
1	98	65	70	71	52	76
2	76	71	83 (16)	63 (32)	54 (45)	51 (21)
3	89	79	91 (10)	64 (40)	51 (34)	80 (31)
4	91	97	94	48	63	68

[a] Based on recovery of 2,3,7,8-TCDD$^{13}C_{12}$ internal standard added before extractions.

TABLE 12. DUPLICATE ANALYSIS RESULTS[a]

Test No.	Analyte	Cyclone Catch, ng/gram	Scrubber Water, ng/liter	Quench Water ng/liter	Spent Carbon Feed, ng/gram
2	2,3,7,8-TCDD	<0.04 (83)	0.07 (63)	<0.02 (54)	<0.02 (51)
		<0.03 (16)	0.04 (32)	<0.02 (45)	<0.03 (21)
	Total TCDD	0.20 (83)	0.21 (63)	<0.04 (54)	<0.04 (51)
		0.16 (16)	0.13 (32)	<0.04 (45)	<0.06 (21)
	2,3,7,8-TCDF	2.71 (83)	<0.01 (63)	<0.06 (54)	<0.04 (51)
		1.00 (16)	<0.01 (32)	<0.06 (45)	<0.07 (21)
	Total TCDF	3.33 (83)	<0.02 (63)	<0.12 (54)	<0.08 (51)
		1.79 (16)	<0.02 (32)	<0.12 (45)	<0.14 (21)
3	2,3,7,8-TCDD	0.01 (91)	0.06 (64)	<0.02 (51)	<0.02 (80)
		0.02 (10)	0.04 (40)	<0.02 (34)	<0.02 (31)
	Total TCDD	4.14 (91)	0.12 (64)	<0.04 (51)	<0.04 (80)
		4.00 (10)	0.14 (40)	<0.04 (34)	<0.04 (31)
	2,3,7,8-TCDF	1.71 (91)	<0.01 (64)	<0.03 (51)	<0.01 (80)
		0.60 (10)	<0.02 (40)	<0.04 (34)	<0.04 (31)
	Total TCDF	2.14 (91)	<0.02 (64)	<0.06 (51)	<0.02 (80)
		1.10 (10)	<0.04 (40)	<0.08 (34)	<0.08 (31)

(a) Extraction efficiencies for determinations based on recovery of 2,3,7,8-TCDD-$^{13}C_{12}$ internal standard are shown in parentheses.

TABLE 13. BLANK ANALYSIS RESULTS

Blank	TCDD Concentration, ng/sample		TCDF Concentration, ng/sample	
	2,3,7,8	Total	2,3,7,8	Total
Mod. Method 5 Filter and 250 ml 50:50 MeOH/MeCl Rinse (Field blanks)[a]	<0.01	<0.02	<0.05	<0.1
22 g XAD-2 and 200 ml MeCl Rinse (Field blanks)[a]	<0.08	<0.16	<0.07	<0.14
1 L benzene used for extractions (laboratory blank)[b]	<0.008	<0.02	<0.005	<0.01
1 L methylene chloride used for extractions (laboratory blank)[b]	<0.007	<0.02	<0.005	<0.01

(a) Composition and quantities of reagents and materials are the same as those used for the actual samples.

(b) 250 mL of benzene or methylene chloride used for Soxhlet extractions. 300 mL of benzene used for liquid-liquid extractions.

ACCURACY AND PRECISION

Estimates of accuracy and precision of the TCDD measurements were made by spiking each type of sample from Tests 1 and 4 with known quantities of 1,2,3,4-TCDD. The 1,2,3,4-TCDD isomer was selected for spiking because previous work (5,6) has shown that 1,2,3,4-TCDD is normally not produced by combustion sources. The XAD-2 traps were spiked prior to conducting the sampling and thus would reflect errors in the TCDD measurements due to volatilization or decomposition losses during sampling as well as errors occuring during the analysis steps. The remainder of the samples were spiked after sampling and before the extraction step; therefore these samples would indicate only errors associated with the analytical portion of the measurement.

Duplicate analyses of the cyclone catch, scrubber water, quench water, and spent carbon feed samples from Tests 2 and 3 were also performed to provide an estimate of the precision of the TCDD and TCDF measurements. Duplicate analyses of the Method 5 samples were not performed because the samples (filter and XAD-2) could not be accurately subdivided.

The accuracy and precision estimates obtained from the spike and duplicate analyses are shown in Tables 14, 15, and 16. The estimations of accuracy and precision for each sample type based on the 1,2,3,4-TCDD spike data are presented in Table 14. The accuracy values reported in the table were calculated by the equation:

$$\text{Accuracy, } \% = \frac{(X-T)\ 100}{T}$$

where X is the quantity of 1,2,3,4-TCDD determined by analysis along with the native TCDD isomers and T is the quantity of spike added either before sampling (XAD-2 traps) or before extraction of the samples.

The precision estimates were obtained by calculating the absolute standard deviation (n-1) for the two determinations for each sample type. The precision estimates are also given as the relative standard deviation (RSD) which was calculated by the equation

$$\text{RSD, } \% = \frac{\sigma 100}{\bar{x}}$$

where σ is the absolute standard deviation and $\bar{x}$ is the average quantity of 1,2,3,4-TCDD found for the two determinations.

The precision estimates obtained from the duplicate TCDD and TCDF analyses are shown in Tables 15 and 16, respectively. The absolute standard deviations (n-1) were calculated from the two determinations of the 2,3,7,8- and total TCDD and the 2,3,7,8- and total TCDF for each sample type. The RSD was calculated as described previously.

TABLE 14. 1,2,3,4-TCDD SPIKE DATA

Sample Type[a]	Test No. 1			Test No. 4			Statistical Estimates			
	Spike Added, ng	Spike Found, ng	Accuracy, %	Spike Added, ng	Spike Found, ng	Accuracy, %	Avg. Spike Found, ng	Average Accuracy, %	Std. Dev. (σ), ng	RSD, %
Mod. Method 5 Filter/Rinse	4.60	5.06	10.0	4.60	4.28	-7.0	4.67	1.5	0.55	11.8
XAD-2	1.20	1.18	- 1.7	1.20	1.08	-10.0	1.13	-5.9	0.07	6.2
Cyclone Catch	20.80	21.00	1.0	20.80	23.71	14.0	22.36	7.5	1.92	8.6
Scrubber Water	4.20	3.57	-15.0	4.20	3.24	-22.9	3.41	-19.0	0.23	6.7
Quench Water	4.20	4.04	- 3.8	4.20	3.70	-11.9	3.87	- 7.9	0.24	6.2
Spent Carbon Feed	4.20	3.26	-22.4	4.20	4.40	4.8	3.83	- 8.8	0.81	21.1

(a) The XAD-2 traps were spiked prior to sampling; all other samples spiked prior to extraction.

TABLE 15. DUPLICATE TCDD ANALYSIS DATA

Test No.	Sample Type	2,3,7,8 TCDD Data				Total TCDD Data			
		Sample	Average	Std. Dev.(σ)	RSD, %	Sample	Average	Std. Dev.(σ)	RSD, %
2	Cyclone Catch	<0.04 ng/g <0.03 ng/g	--	--	--	0.20 ng/g 0.16 ng/g	0.18 ng/g	0.03 ng/g	17
3	Cyclone Catch	0.01 ng/g 0.02 ng/g	0.02 ng/g	0.01 ng/g	50	4.14 ng/g 4.00 ng/g	4.07 ng/g	0.10 ng/g	2
2	Scrubber Water	0.07 ng/l 0.04 ng/l	0.06 ng/L	0.02 ng/L	33	0.21 ng/l 0.13 ng/l	0.17 ng/L	0.06 ng/L	35
3	Scrubber Water	0.06 ng/l 0.04 ng/l	0.05 ng/L	0.01 ng/L	20	0.12 ng/l 0.14 ng/l	0.13 ng/L	0.01 ng/L	8

TABLE 16. DUPLICATE TCDF ANALYSIS DATA

Test No.	Sample Type	2,3,7,8-TCDF Data				Total TCDF Data			
		Sample, ng/g	Average ng/g	Std. Dev.(σ), ng/g	RSD, %	Sample ng/g	Average, ng/g	Std. Dev.(σ), ng/g	RSD, %
2	Cyclone Catch	2.71 1.00	1.86	1.21	65	3.33 1.79	2.56	1.09	43
3	Cyclone Catch	1.71 0.60	1.16	0.78	67	2.14 1.10	1.62	0.74	46

In general, the accuracy and precision estimates obtained from the 1,2,3,4-TCDD spike data are well within the quality assurance objectives for the program. On the other hand, greater variability was observed in the duplicate TCDD and TCDF analyses performed on the cyclone catch and scrubber water samples. The poorer precision estimated from some of the duplicate TCDD and TCDF analyses is due to the increased variability associated with measurement of concentrations approaching the detection limit. Also, low extraction efficiencies were obtained for the initial set of samples. Thus, quantitation errors were greater because of the difficulty in measuring the small native isomers and the 2,3,7,8-TCDD-$^{13}C_{12}$ internal standard peak areas. Higher extraction efficiencies and presumably more accurate data were obtained for the second set of samples by modifying the extraction procedure as described previously.

COMPLETENESS

Valid results are reported for all samples for which collection and analyses were planned during this study. Therefore, the completeness objective of 100 percent was achieved.

AUDITS

A systems audit of the sampling program and the analytical procedures and facilities employed in the study was performed by Dr. Bruce Cobley of S-CUBED. A copy of the audit report is given in Appendix D.

DATA RECORDS

Data accumulated during this study are recorded in Battelle-Columbus Laboratories (BCL) Laboratory Record Books Nos. 37900, 37901, and 37534. The mass spectral data are recorded on magnetic tapes and stored in the BCL Mass Spectrometry Laboratory tape library.

The magnetic tape records will be retained for three years following completion of the study. The Laboratory Records Books will be retained in a permanent file maintained by Battelle-Columbus Laboratories.

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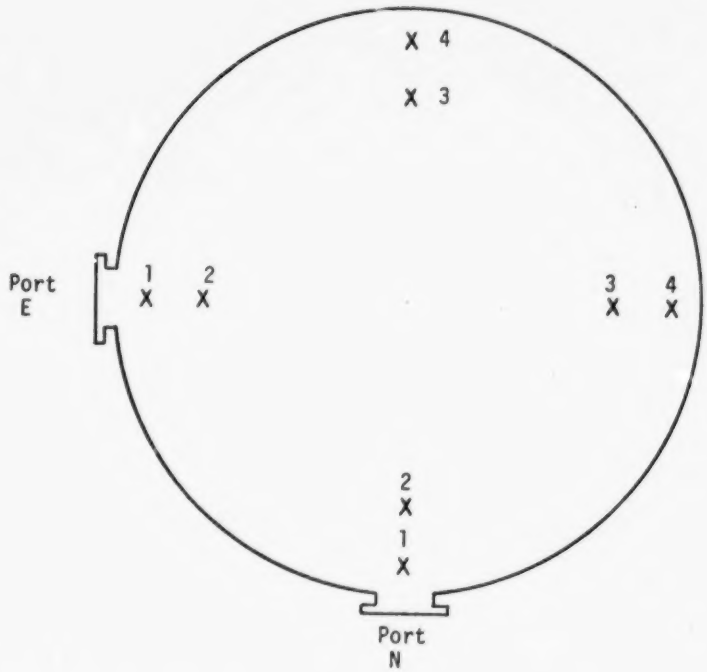
APPENDIX A

FIELD SAMPLING AND SAMPLE RECOVERY DATA

The field data recorded during collection of samples from the Cincinnati Waterworks reactivator unit are presented in Figures A-1 through A-31. The order used to present the figures are:

- Stack gas velocity and temperature traverse data
- Modified Method 5 System operating data
- Fyrite stack gas measurements for CO_2 and O_2
- Modified Method 5 sample recovery data
- Scrubber water, quench water, cyclone catch, and spent carbon feed sampling data
- Natural gas sampling data.



Stack ID = 8.25 in (21 cm) Static Pressure = -0.13 in. (-0.33 cm) H_2O

Traverse Point	Distance from Port on ID, in (cm)	Velocity Head (ΔP) in H_2O (cm H_2O)	Stack Temperature (T_s) °F	(°C)
N-1	0.55 (1.40)	0.25 (0.64)	111	(44)
N-2	2.06 (5.23)	0.28 (0.71)	111	(44)
N-3	6.19 (15.72)	0.29 (0.73)	111	(44)
N-4	7.70 (19.56)	0.26 (0.66)	111	(44)
E-1	0.55 (1.40)	0.24 (0.61)	111	(44)
E-2	2.06 (5.23)	0.28 (0.71)	111	(44)
E-3	6.19 (15.72)	0.31 (0.79)	111	(44)
E-4	7.70 (19.56)	0.29 (0.73)	111	(44)
Average		0.28 (0.71)	111	(44)

Figure A-1. Stack velocity and temperature traverse data

Plant/Address: Cincinnati Waterworks Plant
Date: 8-30-82
Sampling Location: Regenerator Stack
Source Code: CW
Run Number: BCL-CW-083082-MM5-1
Operator: Kohler
Ambient Temperature, °F: 83 F
Barometric Pressure, in. Hg: 29.51" Hg
Static Pressure, in. Hg: 29.50" Hg

Pre-Test Leak Rate: 0.004 cfm @ -15 in. Hg
Post-Test Leak Rate: 0.008 cfm @ -20 in. Hg

Pitot tubes checked at >2" H_2O before test

Probe Length and Type: 44" Borosilicate S/N 569-44-1
Nozzle S/N: 0.303 in. Nozzle ID: 303-1
Assumed Moisture, %: 9
XAD-2 Module S/N: ST-1
Meter Box S/N: 1971 Oven S/N: B
Meter $\Delta H_@$: 1.91
C Factor: 1.02
Probe Heater Setting: 84%
Oven Setting: 250 F
Reference ΔP: 0.249
Calculations Checked By: Howes Date:

Traverse Point Number	Sampling Time, min	Clock Time (24-hr Clock)	Gas Meter Reading, ft^3	Velocity Head, in. H_2O	Orifice Pressure Differential, in. H_2O Desired	Orifice Pressure Differential, in. H_2O Actual	Stack Temperature, °F	Dry Gas Meter Temperature Inlet, °F	Dry Gas Meter Temperature Outlet, °F	Pump Vacuum, in. Hg	Oven Temperature, °F	Impinger Temperature, °F	Sorbent Trap Temperature, °F	Probe Temperature, °F
N-4	0	1323	863.897	0.28	2.10	2.10	109	84	84	15.0	240	49	42	189
N-4	10	1333	871.600	0.28	2.10	2.10	109	102	86	15.5	250	51	42	203
N-4	20	1343	878.990	0.26	1.92	1.92	108	110	90	15.0	249	53	45	213
N-4	30	1353	886.245	0.26	1.92	1.92	108	114	94	14.5	250	56	48	221
N-4	40	1403	893.490	0.31	2.3	2.3	108	116	98	18.0	249	59	52	221
N-4	50	1413	901.400	0.30	2.25	2.25	108	118	99	18.0	250	55	43	221
N-4	60	1423	909.370	0.29	2.15	2.15	108	120	100	17.5	244	52	41	233
N-4	70	1433	917.330	0.28	2.10	2.10	108	120	101	17.5	249	51	42	239
N-4	80	1443	925.175	0.28	2.10	2.10	108	120	102	16.5	249	51	43	246
N-4	90	1453	933.030	0.30	2.25	2.25	108	122	102	18.0	250	54	42	247
N-4	100	1503	941.100	0.30	2.25	2.25	108	121	103	18.5	249	54	42	248
N-4	110	1513	949.250	0.30	2.25	2.25	108	121	103	18.5	247	54	44	245
N-4	120	1523	957.380	0.29	2.15	2.15	108	122	104	17.5	250	55	46	248
N-4	130	1533	965.410	0.29	2.15	2.15	108	122	103	17.5	248	55	47	252
N-4	140	1543	973.850	0.29	2.15	2.15	107	122	103	17.5	248	56	45	252
N-4	150	1553	981.395	0.28	2.10	2.10	107	122	103	17.0	248	57	46	253
N-4	160	1603	989.300	0.28	2.10	2.10	107	122	103	17.0	250	54	44	255
N-4	170	1613	997.240	0.27	2.00	2.00	107	122	103	15.0	248	49	46	252
N-4	180	1623	1004.85	0.27	2.00	2.00	107	121	104	15.0	250	48	47	259
Total														
Average														

Figure A-2. Modified method 5 stack sampling data - Test No. 1.

Plant/Address: Cincinnati Water Works Plant
Date: 8-30-82
Sampling Location: Regenerator Stack
Source Code: CW
Run Number: BCL-CW-083082-MM5-1
Operator: Kohler
Ambient Temperature, °F: 83 F
Barometric Pressure, in. Hg: 29.51" Hg
Static Pressure, in. Hg: 29.50" Hg

Pre-Test Leak Rate: 0.004 cfm @ -15 in. Hg
Post-Test Leak Rate: 0.008 cfm @ -20 in. Hg

Pitot tubes checked at >2" H_2O before test

Probe Length and Type: 44" Borosilicate S/N 569-44-1
Nozzle S/N: 0.303 in. Nozzle ID: 303-1
Assumed Moisture, %: 9
XAD-2 Module S/N: ST-1
Meter Box S/N: 1971 Oven S/N: B
Meter $\Delta H_@$: 1.91
C Factor: 1.02
Probe Heater Setting: 84%
Oven Setting: 250 F
Reference ΔP: 0.249
Calculations Checked By: Howes Date:

Traverse Point Number	Sampling Time, min	Clock Time (24-hr Clock)	Gas Meter Reading, ft^3	Velocity Head, in. H_2O	Orifice Pressure Differential, in. H_2O Desired	Orifice Pressure Differential, in. H_2O Actual	Stack Temperature, °F	Dry Gas Meter Temperature Inlet, °F	Dry Gas Meter Temperature Outlet, °F	Pump Vacuum, in. Hg	Oven Temperature, °F	Impinger Temperature, °F	Sorbent Trap Temperature, °F	Probe Temperature, °F
N-4	190	1633	1012.34	0.27	2.0	2.0	107	120	103	15	248	48	47	258
N-4	200	1643	1019.81	0.27	2.0	2.0	107	120	103	15	250	50	46	258
N-4	210	1653	1027.29	0.27	2.0	2.0	107	120	103	15	250	51	46	258
N-4	220	1703	1034.75	0.27	2.0	2.0	107	119	103	15	250	52	44	257
N-4	230	1713	1042.28	0.27	2.0	2.0	108	119	103	15	250	55	43	255
N-4	240	1723	1049.765		END OF TEST									
			PITOT TUBES CHECKED AFTER TEST @ >2" H_2O											
Total	240		185.868	$(\sqrt{\Delta P})$AVG										
Average				0.5306		2.10	107.7	108.7			249		45	241

Figure A-2. Modified method 5 stack sampling data - Test No. 1 (continued).

Plant/Address: Cincinnati Water Works Plant
Date: 8/31/82
Sampling Location: Regenerator Stack
Source Code: CW
Run Number: BCL-CW-083182-MM5-2
Operator: Kohler
Ambient Temperature, °F: 76 F
Barometric Pressure, in. Hg: 29.50" Hg
Static Pressure, in. Hg: 29.49" Hg

Pre-Test Leak Rate
0.006 cfm @ -20 in. Hg
Post-Test Leak Rate
___ cfm @ ___ in. Hg

Pitot Tubes Checked at >2" H_2O

Probe Length and Type: 44" Borosilicate S/N
Nozzle S/N: 0.303" Nozzle ID
Assumed Moisture, %: 9
XAD-2 Module S/N: ST-2
Meter Box S/N: 1971 Oven S/N: B
Meter $\Delta H_@$: 1.91
C Factor: 1.04
Probe Heater Setting: 95%
Oven Setting: 250
Reference ΔP: 0.22
Calculations Checked By: Howes Date

Traverse Point Number	Sampling Time, min	Clock Time (24-hr Clock)	Gas Meter Reading, ft^3	Velocity Head, in. H_2O	Orifice Pressure Differential, in. H_2O Desired	Orifice Pressure Differential, in. H_2O Actual	Stack Temperature, °F	Dry Gas Meter Temperature Inlet, °F	Dry Gas Meter Temperature Outlet, °F	Pump Vacuum, in. Hg	Oven Temperature, °F	Impinger Temperature, °F	Sorbent Trap Temperature, °F	Probe Temperature, °F
N-4	0	0847	053.902	0.28	2.35	2.35	108	81	81	18	247	53	45	259
N-4	10	0857	062.11	0.30	2.50	2.50	108	97	83	18	245	51	41	233
N-4	20	0907	070.45	0.29	2.45	2.45	108	113	90	19	244	50	45	232
N-4	30	0917	078.86	0124	2.02	2.02	108	117	94	15	246	51	46	235
N-4	40	0927	086.47	0.23	1.90	1.90	105	118	97	13.5	243	52	47	242
N-4	50	0937	094.00	0.26	2.18	2.18	107	118	100	15.0	243	51	41	241
N-4	60	0947	101.57	0.29	2.45	2.45	109	119	101	18.2	252	48	40	236
N-4	70	0957	109.98	0.31	2.60	2.60	108	122	102	20.0	251	50	41	238
N-4	80	1007	118.64	0.29	2.45	2.45	106	123	103	19.0	253	53	43	238
N-4	90	1017	127.20	0.23	1.90	1.90	104	124	104	13.2	250	55	45	251
N-4	100	1027	134.57	0.25	2.10	2.10	106	122	104	14.8	251	56	47	254
N-4	110	1037	142.19	0.28	2.35	2.35	109	122	105	17.0	251	53	41	250
N-4	120	1047	150.31	0.31	2.60	2.60	110	124	107	20.5	250	52	41	247
N-4	130	1057	159.03	0.33	2.80	2.80	110	124	105	22.5	251	55	43	249
N-4	140	1107	167.93	0.32	2.70	2.70	110	124	106	22.5	250	57	45	246
N-4	150	1117	176.80	0.31	2.60	2.60	109	125	107	21.5	250	59	47	242
N-4	160	1127	185.55	0.29	2.45	2.45	109	128	107	19.0	253	55	42	246
N-4	170	1137	194.07	0.28	2.35	2.35	109	129	108	18.0	252	53	44	247
N-4	180	1147	202.40	0.28	2.35	2.35	109	129	108	18.0	251	54	45	250
Total														
Average														

Figure A-3. Modified method 5 stack sampling data - Test No. 2.

Plant/Address: Cincinnati Water Works Plant
Date: 8/31/82
Sampling Location: Regenerator Stack
Source Code: CW
Run Number: BCL-CW-083182-MM5-2
Operator: Kohler
Ambient Temperature, °F: 76 F
Barometric Pressure, in. Hg: 29.50" Hg
Static Pressure, in. Hg: 29.49" Hg

Pre-Test Leak Rate: 0.006 cfm @ -20 in. Hg
Post-Test Leak Rate: 0.006 cfm @ -23 in. Hg

Pitot Tubes Checked at >2" H_2O

Probe Length and Type: 44" Borosilicate S/N
Nozzle S/N: 0.303" Nozzle ID
Assumed Moisture, %: 9%
XAD-2 Module S/N: ST-2
Meter Box S/N: 1971 Oven S/N: B
Meter $\Delta H_@$: 1.91
C Factor: 1.04
Probe Heater Setting: 95%
Oven Setting: 250 F
Reference ΔP: 0.22
Calculations Checked By: Howes Date

Traverse Point Number	Sampling Time, min	Clock Time (24-hr Clock)	Gas Meter Reading, ft^3	Velocity Head, in. H_2O	Orifice Pressure Differential, in. H_2O Desired	Orifice Pressure Differential, in. H_2O Actual	Stack Temperature, °F	Dry Gas Meter Temperature Inlet, °F	Dry Gas Meter Temperature Outlet, °F	Pump Vacuum, in. Hg	Oven Temperature, °F	Impinger Temperature, °F	Sorbent Trap Temperature, °F	Probe Temperature, °F
N-4	190	1157	210.80	0.28	235	2.35	110	128	109	18.2	252	55	45	251
N-4	200	1207	218.98	0.31	2.60	2.60	110	128	109	20.0	251	54	44	251
N-4	310	1217	227.64	0.31	2.60	2.60	111	128	109	21.0	252	56	43	248
N-4	220	1227	236.31	0.31	2.60	2.60	110	128	109	21.0	252	56	46	250
N-4	230	1237	244.98	0.30	2.50	2.50	110	129	109	21.0	252	56	47	250
N-4	240	1247	253.608				END OF TEST							
				PITOT TUBES CHECKED AT >4" H_2O										
Total	240		204.706	$(\sqrt{\Delta P})Av$										
Average				0.5348		2.41	108.5	111.6			250		44	245

Figure A-3. Modified method 5 stack sampling data - Test No. 2 (continued).

Plant/Address: Cincinnati Water Works Plant
Date: 8-31-82
Sampling Location: Regenerator Stack
Source Code: CW
Run Number: BCL-CW-083182-MM5-3
Operator: Kohler
Ambient Temperature, °F: 85 F
Barometric Pressure, in. Hg: 29.47" Hg
Static Pressure, in. Hg: 29.46" Hg

Pre-Test Leak Rate 22.5
0.003 cfm @ ___ in. Hg
Post-Test Leak Rate
0.002 cfm @ -15 in. Hg

Pitot tubes checked at >2" H_2O - OK

Probe Length and Type: 44" Borosilicate S/N ___
Nozzle S/N: 0.303 in. Nozzle ID ___
Assumed Moisture, %: 9%
XAD-2 Module S/N: ST-3
Meter Box S/N: 1971 Oven S/N: B
Meter $\Delta H_@$: 1.91
C Factor: 1.04
Probe Heater Setting: 83%
Oven Setting: 250 F
Reference ΔP: 0.22
Calculations Checked By: Howes Date ___

1555 Hours Checked ΔP
NBS Pitot Tube 0.18

Traverse Point Number	Sampling Time, min	Clock Time (24-hr Clock)	Gas Meter Reading, ft^3	Velocity Head, in. H_2O	Orifice Pressure Differential, in. H_2O Desired	Orifice Pressure Differential, in. H_2O Actual	Stack Temperature, °F	Dry Gas Meter Temperature Inlet, °F	Dry Gas Meter Temperature Outlet, °F	Pump Vacuum, in. Hg	Oven Temperature, °F	Impinger Temperature, °F	Sorbent Trap Temperature, °F	Probe Temperature, °F
N-4	0	1443	254.112	0.18	1.52	1.52	107	92	92	9.5	253	60	47	314
N-4	10	1453	260.745	0.19	1.60	1.60	105	104	93	10	252	58	46	297
N-4	20	1503	267.43	0.20	1.70	1.70	105	112	97	10.5	252	62	47	259
N-4	30	1513	274.46	0.22	1.85	1.85	106	117	102	11.0	249	55	41	255
N-4	40	1523	280.55	0.19	1.60	1.60	105	118	102	10	252	54	41	258
N-4	50	1533	288.32	0.17	1.45	1.45	104	119	104	9.2	250	54	43	258
N-4	60	1543	294.78	0.16	1.35	1.35	103	119	106	8.8	251	56	44	254
N-4	70	1553	301.70	0.16	1.35	1.35	103	119	106	8.8	251	55	45	253
N-4	80	1603	307.36	0.12	1.03	1.03	102	118	107	7.5	263	55	47	258
N-4	90	1613	313.08	0.12	1.03	1.03	102	117	107	7.5	264	52	42	260
N-4	100	1623	318.695	0.13	1.12	1.12	104	116	107	7.5	256	50	42	256
N-4	110	1633	324.50	0.15	1.28	1.28	104	117	107	8.3	255	50	43	255
N-4	120	1643	330.61	0.17	1.45	1.45	104	118	107	9.2	253	52	45	252
N-4	130	1653	337.04	0.13	1.12	1.12	104	118	108	8.0	255	54	46	250
N-4	140	1703	343.00	0.12	1.03	1.03	104	118	108	7.5	253	57	47	257
N-4	150	1713	348.685	0.12	1.03	1.03	103	117	108	7.5	253	53	41	255
N-4	160	1723	354.34	0.12	1.03	1.03	103	117	108	7.5	253	51	41	253
N-4	170	1733	360.00	0.09	0.83	0.83	103	117	108	6.5	255	50	43	254
N-4	180	1743	365.23	0.14	1.20	1.20	105	116	108	8.0	254	51	40	255
Total														
Average														

Figure A-4. Modified method 5 stack sampling data - Test No. 3.

Plant/Address: Cincinnati Water Works Plant
Date: 8-31-82
Sampling Location: Regenerator Stack
Source Code: CW
Run Number: BCL-CW-083182-MM5-3
Operator: Kohler
Ambient Temperature, °F: 85 F
Barometric Pressure, in. Hg: 29.47" Hg
Static Pressure, in. Hg: 29.46" Hg

Pre Test Leak Rate: 0.003 cfm @ -22.5 in. Hg
Post Test Leak Rate: 0.002 cfm @ -15 in. Hg

Pitot tubes checked at >2" H_2O - OK

Probe Length and Type: 44" Borosilicate S/N
Nozzle S/N: 0.303 in. Nozzle ID
Assumed Moisture, %: 9%
XAD-2 Module S/N: ST-3
Meter Box S/N: 1971 Oven S/N: B
Meter $\Delta H_@$: 1.91
C Factor: 1.04
Probe Heater Setting: 83%
Oven Setting: 250 F
Reference ΔP: 0.22
Calculations Checked By: Howes Date

Traverse Point Number	Clock Time (24-hr Clock) / Sampling Time, min		Gas Meter Reading, ft^3	Velocity Head, in. H_2O	Orifice Pressure Differential, in. H_2O		Stack Temperature, °F	Dry Gas Meter Temperature		Pump Vacuum, in. Hg	Oven Temperature, °F	Impinger Temperature, °F	Sorbent Trap Temperature, °F	Probe Temperature, °F
					Desired	Actual		Inlet, °F	Outlet, °F					
N-4	186	1749	-	0.18	1.53	1.53	-	-	-	-	-	-	-	-
N-4	190	1753	371.37	0.20	1.70	1.70	105	118	108	10.5	256	52	40	238
N-4	200	1803	378.25	0.23	1.92	1.92	106	120	108	11.5	251	54	42	248
N-4	205	1808	-	0.26	2.20	2.20	-	-	-	14.5	-	-	-	-
N-4	210	1813	385.78	0.25	2.10	2.10	108	123	108	13.0	251	58	44	249
N-4	215	1818	-	0.20	1.70	1.70	-	-	-	12.5	-	-	-	-
N-4	220	1823	393.22	0.13	1.12	1.12	103	122	108	8.0	250	60	49	264
N-4	230	1833	399.16	0.12	1.03	1.03	103	119	108	7.5	250	57	43	253
N-4	240	1843	404.822			END OF TEST								
				PITOT TUBES CHECKED AT >2" H_2O - OK										
Total	240		150.710	$(\sqrt{\Delta P})$AVG										
Average				0.4025		1.40	104.2	110.8			254		44	258

Figure A-4. Modified method 5 stack sampling data - Test No. 3 (continued).

Plant/Address: Cincinnati Waterworks Plant
Date: 9-1-82
Sampling Location: Regenerator Stack
Source Code: CW
Run Number: BCL-CW-090183-MM5-4
Operator: Kohler
Ambient Temperature, °F: 78
Barometric Pressure, in. Hg: 29.43
Static Pressure, in. Hg: 29.42

Pre-Test Leak Rate
0.004 cfm @ -20 in. Hg
Post-Test Leak Rate
0.002 cfm @ -23 in. Hg

Pitot tubes checked at >2" H_2O

Probe Length and Type: 44" Borosilicate S/N
Nozzle S/N: Nozzle ID: 0.303 in.
Assumed Moisture, %: 9%
XAD-2 Module S/N: ST-4
Meter Box S/N: 1971 Oven S/N: B
Meter $\Delta H_@$: 1.91
C Factor: 1.04
Probe Heater Setting: 90%
Oven Setting: 250°F
Reference ΔP: 0.22
Calculations Checked By: Howes Date:

Traverse Point Number	Sampling Time, min	Clock Time (24-hr Clock)	Gas Meter Reading, ft^3	Velocity Head, in. H_2O	Orifice Pressure Differential, in. H_2O Desired	Orifice Pressure Differential, in. H_2O Actual	Stack Temperature, °F	Dry Gas Meter Temperature Inlet, °F	Dry Gas Meter Temperature Outlet, °F	Pump Vacuum, in. Hg	Oven Temperature, °F	Impinger Temperature, °F	Sorbent Trap Temperature, °F	Probe Temperature, °F
N-4	0	0915	410.081	0.30	2.55	2.25	111	87	87	23	263	75	72	256
N-4	10	0925	417.90	0.34	2.90	2.25	113	108	92	23	261	50	46	253
N-4	20	0935	425.79	0.39	3.30	2.25	114	115	96	23	263	50	46	252
N-4	30	0945	433.61	0.40	3.40	2.25	114	119	99	23	263	50	47	252
N-4	40	0955	441.49	0.27	2.25	2.25	104	120	102	23	262	47	45	252
N-4	45	1000	445.46	0.19	1.62	1.62	--	--	--	--	--	--	--	--
N-4	50	1005	448.93	0.19	1.62	1.62	103	123	103	15	261	47	47	253
N-4	60	1015	455.78	0.26	2.20	2.20	110	122	104	22	263	48	45	244
N-4	70	1025	463.62	0.32	2.70	2.25	111	122	105	23	264	51	45	253
N-4	80	1035	471.54	0.35	2.98	2.25	114	122	105	23	262	51	46	252
N-4	90	1045	479.41	0.35	2.98	2.25	114	123	105	23	262	52	46	253
N-4	100	1055	487.28	0.35	2.98	2.25	114	123	105	23	260	53	45	254
N-4	110	1105	495.12	0.35	2.98	2.20	113	124	106	23	263	55	46	250
N-4	120	1115	502.94	0.35	2.98	2.20	112	124	106	23	263	52	45	252
N-4	130	1125	510.78	0.34	2.90	2.20	112	124	106	23	262	52	46	252
N-4	140	1135	518.58	0.34	2.90	2.20	112	124	106	23	262	52	48	255
N-4	150	1145	526.35	0.32	2.70	2.20	112	124	107	23	263	54	45	252
N-4	160	1155	534.16	0.32	2.70	2.20	112	124	107	23	262	51	45	252
N-4	170	1205	542.00	0.32	2.70	2.20	113	125	107	23	261	50	45	253
Total														
Average														

Figure A-5. Modified method 5 stack sampling data - Test No. 4.

Plant/Address: Cincinnati Waterworks Plant
Date: 9-1-82
Sampling Location: Regenerator Stack
Source Code: CW
Run Number: BCL-CW-090182-MM5-4
Operator: Kohler
Ambient Temperature, °F: 78 F
Barometric Pressure, in. Hg: 29.43
Static Pressure, in. Hg: 29.42

Pre-Test Leak Rate: 0.004 cfm @ -20 in. Hg
Post-Test Leak Rate: 0.002 cfm @ -23 in. Hg

Pitot tubes checked

Probe Length and Type: 44" Borosilicate S/N
Nozzle S/N: Nozzle ID: 0.303 in.
Assumed Moisture, %: 9%
XAD-2 Module S/N: ST-4
Meter Box S/N: 1971 Oven S/N: B
Meter $\Delta H_@$: 1.91
C Factor: 1.04
Probe Heater Setting: 90%
Oven Setting: 250°F
Reference ΔP: 0.22
Calculations Checked By: Howes Date:

Traverse Point Number	Sampling Time, min	Clock Time (24-hr Clock)	Gas Meter Reading, ft^3	Velocity Head, in. H_2O	Orifice Pressure Differential, in. H_2O Desired	Orifice Pressure Differential, in. H_2O Actual	Stack Temperature, °F	Dry Gas Meter Temperature Inlet, °F	Dry Gas Meter Temperature Outlet, °F	Pump Vacuum, in. Hg	Oven Temperature, °F	Impinger Temperature, °F	Sorbent Trap Temperature, °F	Probe Temperature, °F
N-4	180	1215	549.78	0.32	2.70	2.20	113	125	108	23	262	51	47	254
N-4	190	1225	557.54	0.32	2.70	2.20	113	125	108	23	262	54	48	252
N-4	200	1235	565.29	0.32	2.70	2.20	112	125	108	23	260	54	47	252
N-4	210	1245	573.04	0.32	2.70	2.20	112	125	108	23	261	55	47	251
N-4	220	1255	580.77	0.32	2.70	2.20	112	125	108	23	262	56	48	252
N-4	230	1305	588.47	0.32	2.70	2.20	112	125	108	23	261	57	49	253
N-4	240	1315	596.17		END OF TEST									
					Pitot Tubes Checked at >2" H_2O									
Total	240		186.09	$(\sqrt{\Delta P})$Avg										
Average				05628		2.18	111.8	112.5			262		47	252

Figure A-5. Modified method 5 stack sampling data - Test No. 4 (continued).

Date 8/30/83 Plant Cincinnati Waterworks

Run No. BCL-CW-083082-MM5-1 Sampling Location Carbon Regenerator Stack

Performed by J. Howes Sample Type Grab

Time	CO_2 Concentration, % by Volume, dry	O_2 Concentration, % by Volume, dry	N_2 Concentration,* % by Volume, dry
1413	12.0	6.5	
1418	13.0	6.0	
1502	12.8	4.0	
1605	13.0	3.5	
1702	13.0	3.0	
Averages	12.76	4.6	82.64

Figure A-6. Fyrite analysis of stack gas - Test No. 1.

Date 8/31/82

Run No. BCL-CW-083182-MM5-2

Performed by J. Howes

Plant Cincinnati Waterworks

Sampling Location Carbon Regenerator Stack

Sample Type Grab

Time	CO_2 Concentration, % by Volume, dry	O_2 Concentration, % by Volume, dry	N_2 Concentration,* % by Volume, dry
0938	13.3	3.0	
1054	12.5	3.0	
1155	13.0	4.0	
1230	13.0	4.0	
Averages	12.95	3.5	83.55

Figure A-7. Fyrite analysis of stack gas - Test No. 2.

Date 8/31/82

Run No. BCL-CW-083182-MM5-3

Performed by J. Howes

Plant Cincinnati Waterworks

Sampling Location Carbon Regenerator Stack

Sample Type Grab

Time	CO_2 Concentration, % by Volume, dry	O_2 Concentration, % by Volume, dry	N_2 Concentration,* % by Volume, dry
1521	12.2	4.0	
1620	12.0	3.5	
1735	11.0	4.5	
1820	12.0	4.0	
Averages	11.8	4.0	84.2

Figure A-8. Fyrite analysis of stack gas - Test No. 3.

Date 9/1/82 Plant Cincinnati Waterworks

Run No. BCL-CW-090182-MM5-4 Sampling Location Carbon Regenerator Stack

Performed by J. Howes Sample Type Grab

Time	CO_2 Concentration, % by Volume, dry	O_2 Concentration, % by Volume, dry	N_2 Concentration,* % by Volume, dry
1023	13.0**	3.5	
1026	13.6	3.5	
1130	14.0	4.0	
1251	14.5	3.0	
1310	14.0	3.5	
Averages	14.03	3.5	82.47

**CO_2 Fyrite did not zero after measurement; not included in average

Figure A-9. Fyrite analysis of stack gas - Test No. 4.

Plant/Location Cincinnati Waterworks

Sampling Location Carbon Regenerator Stack

Train Type Mod. Method 5 Run No. BCL-CW-083082-MM5-1 Run Date 8/30/82

Recovery Date 8/30/82 Recovered by Kohler/Howes Checked by J. Howes

1.0 CYCLONE CATCH DATA

Cyclone	Sample No.	Tare, g	Gross, g	Net, g
10 μm				
3 μm				
1 μm		N/A		

2.0 FILTER CATCH DATA

Filter Size/Material 3" dia. Pallflex 2500 QAST Quartz Fiber

Filter No.	Sample No.	Tare, g	Gross, g	Net, g
PD-1	BCL-CW-083082-MM5-1-QPF	0.3716	0.3766	0.0050

Filter Total 0.0050 g

Figure A-10. Modified method 5 sample recovery data - Test No. 1.

3.0 CONDENSATE COLLECTION

Total Condensate Collected _____ g

288 ml

4.0 IMPINGER DATA

Impinger No.	Sample No.	Loading	Initial*	Final*	Gain*
1	Discarded	100 ml DI Water	577.9 g	586.0 g	8.1 g
2	"	"	593.9 g	594.6 g	0.7 g
3	"	Empty	520.5 g	524.2 g	3.7 g
4					
5					

Total Gain* 12.5 g

* Indicate whether ml or g

5.0 SILICA GEL WEIGHTS

Initial Weight, g	Final Weight, g	Weight Gain, g
784.4	825.9	41.5

Total Weight Gain 41.5 g

6.0 TOTAL MOISTURE COLLECTED

Total Collected = Condensate, g + Total Imp. Gain, g + Total Silica Gel Gain, g

= 342 g

7.0 SOLID SORBENT TRAP

Solid Sorbent Material Purified XAD-2 (Spiked with 1,2,3,4-TCDD)

Weight Used 22 g g

Trap No. ST-1

Sample No. BCL-CW-083082-MM5-1-XAD

Figure A-10. Modified method 5 sample recovery data - Test No. 1 (continued).

8.0 RINSE SOLUTION DATA

Component	Sample No.	Rinse Composition	Rinse Volume, ml
Front Half	BCL-CW-083082-MM5-1-FHR	MeOH/MeCl 50:50	265
Condenser Mod.	BCL-CW-083082-MM5-1-OMR	MeCl	~ 200
Impinger #1			
Impinger #2			
Impinger #3			
Impinger #4			

Comments: (Indicate condition of filters, solution colors, presence of precipitates, odor, etc.)

* Filter in good condition; no evidence of leakage. Light gray deposit on filter
* Condensate clear
* Impinger solutions clear

FRONT HALF RINSE RESIDUE WEIGHT (Rinse volume = 265 ml)

G 65.5890 g
T 65.5733 g
N 0.0157 g

Blank correction negligible

Net residue wt = 0.0157 g
Net filter wt = 0.0050 g

Total Net Particulate Wt = 0.0207 g

Figure A-10. Modified method 5 sample recovery data - Test No. 1 (continued).

Plant/Location Cincinnati Waterworks

Sampling Location Carbon Regenerator Stack

Train Type Mod. Method 5 Run No. BCL-CW-083182-MM5-2 Run Date 8/31/82

Recovery Date 8/31/82 Recovered by Kohler/Howes Checked by J. Howes

1.0 CYCLONE CATCH DATA

Cyclone	Sample No.	Tare, g	Gross, g	Net, g
10 μm		N/A		
3 μm				
1 μm				

2.0 FILTER CATCH DATA

Filter Size/Material 3" dia. Pallflex 2500 QAST Quartz Fiber

Filter No.	Sample No.	Tare, g	Gross, g	Net, g
PD-2	BCL-CW-083182-MM5-2-QPF	0.3737	0.3809	0.0072

Filter Total 0.0072 g

Figure A-11. Modified method 5 sample recovery data - Test No. 2.

3.0 CONDENSATE COLLECTION

Total Condensate Collected _____ g

319 ml

4.0 IMPINGER DATA

Impinger No.	Sample No.	Loading	Initial*	Final*	Gain*
1	Discarded	100 ml DI Water	594.1 g	598.5 g	4.4 g
2	"	"	578.3 g	582.7 g	4.4 g
3	"	Empty	521.0 g	524.2 g	3.2 g
4					
5					

Total Gain* 12.0 g

* Indicate whether ml or g

5.0 SILICA GEL WEIGHTS

Initial Weight, g	Final Weight, g	Weight Gain, g
769.1	808.5	39.4

Total Weight Gain 39.4 g

6.0 TOTAL MOISTURE COLLECTED

Total Collected = Condensate, g + Total Imp. Gain, g + Total Silica Gel Gain, g

= 370.4 g

7.0 SOLID SORBENT TRAP

Solid Sorbent Material Purified XAD-2

Weight Used 22 g g

Trap No. ST-2

Sample No. BCL-CW-083182-MM5-2-XAD

Figure A-11. Modified method 5 sample recovery data - Test No. 2 (continued).

8.0 RINSE SOLUTION DATA

Component	Sample No.	Rinse Composition	Rinse Volume, ml
Front Half	BCL-CW-083182-MM5-2-FHR	MeOH/MeCl 50:50	248
Condenser Mod.	BCL-CW-083182-MM5-2-OMR	MeCl	~ 200
Impinger #1			
Impinger #2			
Impinger #3			
Impinger #4			

Comments: (Indicate condition of filters, solution colors, presence of precipitates, odor, etc.)

* Filter in good condition; no evidence of leakage. Greyish-black deposit on filter.
* Condensate clear
* Impinger solutions clear

FRONT HALF RINSE RESIDUE WEIGHT (Rinse Volume = 248 ml)

G 65.1330 g
T 65.1205 g
N 0.0125 g

Blank correction negligible

Net residue wt = 0.0125 g
Net filter wt = 0.0072 g

Total Net Particulate Wt = 0.0197 g

Figure A-11. Modified method 5 sample recovery data - Test No. 2 (continued).

Plant/Location Cincinnati Waterworks

Sampling Location Carbon Regenerator Stack

Train Type Mod. Method 5 Run No. BCL-CW-083182-MM5-3 Run Date 8/31/82

Recovery Date 8/31/82 Recovered by Kohler/Howes Checked by J. Howes

1.0 CYCLONE CATCH DATA

Cyclone	Sample No.	Tare, g	Gross, g	Net, g
10 μm		N/A		
3 μm				
1 μm				

2.0 FILTER CATCH DATA

Filter Size/Material 3" dia. Pallflex 2500 QAST Quartz Fiber

Filter No.	Sample No.	Tare, g	Gross, g	Net, g
PD-3	BCL-CW-083182-MM5-3-QPF	0.3749	0.3814	0.0065

Filter Total 0.0065 g

Figure A-12. Modified method 5 sample recovery data - Test No. 3.

3.0 CONDENSATE COLLECTION

Total Condensate Collected _____ g

166 ml

4.0 IMPINGER DATA

Impinger No.	Sample No.	Loading	Initial*	Final*	Gain*
1	Discarded	100 ml DI Water	577.7 g	588.2 g	10.5 g
2	"	"	586.3 g	588.3 g	2.0 g
3	"	Empty	481.5 g	484.6 g	3.1 g
4					
5					

Total Gain* 15.6 g

* Indicate whether ml or g

5.0 SILICA GEL WEIGHTS

Initial Weight, g	Final Weight, g	Weight Gain, g
817.8	853.5	35.7

Total Weight Gain 35.7 g

6.0 TOTAL MOISTURE COLLECTED

Total Collected = Condensate, g + Total Imp. Gain, g + Total Silica Gel Gain, g

= 217.3 g

7.0 SOLID SORBENT TRAP

Solid Sorbent Material Purified XAD-2

Weight Used 22 g g

Trap No. ST-3

Sample No. BCL-CW-083182-MM5-3-XAD

Figure A-12. Modified method 5 sample recovery data - Test No. 3 (continued).

8.0 RINSE SOLUTION DATA

Component	Sample No.	Rinse Composition	Rinse Volume, ml
Front Half	BCL-CW-083182-MM5-3-FHR	MeOH/MeCl 50:50	237
Condenser Mod.	BCL-CW-083182-MM5-3-OMR	MeCl	~200
Impinger #1			
Impinger #2			
Impinger #3			
Impinger #4			

Comments: (Indicate condition of filters, solution colors, presence of precipitates, odor, etc.)

* Filter in good condition; no evidence of leakage. Greyish-black deposit on filter.
* Condensate clear
* Impinger solutions clear

FRONT HALF RINSE RESIDUE WEIGHT (Rinse Volume = 237 ml)

G 65.3232 g
T 65.3235 g
N -0.0003 g

Blank correction negligible

Net residue wt = 0.0000 g
Net filter wt = 0.0065 g

Total Net Particulate Wt = 0.0065 g

Figure A-12. Modified method 5 sample recovery data - Test No. 3 (continued).

Plant/Location Cincinnati Waterworks

Sampling Location Carbon Regenerator Stack

Train Type Mod. Method 5 Run No. BCL-CW-090182-MM5-4 Run Date 9/1/82

Recovery Date 9/1/82 Recovered by Kohler/Howes Checked by J. Howes

1.0 CYCLONE CATCH DATA

Cyclone	Sample No.	Tare, g	Gross, g	Net, g
10 μm		N/A		
3 μm				
1 μm				

2.0 FILTER CATCH DATA

Filter Size/Material 3" dia. Pallflex 2500 QAST Quartz Fiber

Filter No.	Sample No.	Tare, g	Gross, g	Net, g
PD-4	BCL-CW-010182-MM5-4-QPF	0.3735	0.3831	0.0096

Filter Total 0.0096 g

Figure A-13. Modified method 5 sample recovery data - Test No. 4.

3.0 CONDENSATE COLLECTION

Total Condensate Collected _____ g

341 ml

4.0 IMPINGER DATA

Impinger No.	Sample No.	Loading	Initial*	Final*	Gain*
1	Discarded	100 ml DI Water	573.2 g	581.5 g	8.3
2	"	"	581.5 g	581.5 g	0.0
3	"	Empty	484.3 g	486.1 g	1.8
4					
5					

Total Gain* 10.1 g

* Indicate whether ml or g

5.0 SILICA GEL WEIGHTS

Initial Weight, g	Final Weight, g	Weight Gain, g
837.6	875.9	38.3

Total Weight Gain 38.3 g

6.0 TOTAL MOISTURE COLLECTED

Total Collected = Condensate, g + Total Imp. Gain, g + Total Silica Gel Gain, g

= 389.4 g

7.0 SOLID SORBENT TRAP

Solid Sorbent Material Purified XAD-2 spiked with 1,2,3,4-TCDD

Weight Used 22 g g

Trap No. ST-4

Sample No. BCL-CW-090182-MM5-4-XAD

Figure A-13. Modified method 5 sample recovery data - Test No. 4 (continued).

8.0 RINSE SOLUTION DATA

Component	Sample No.	Rinse Composition	Rinse Volume, ml
Front Half	BCL-CW-090182-MM5-4-FMR	MeOH/MeCl 50:50	212
Condenser Mod.	BCL-CW-090182-MM5-4-OMR	MeCl	~ 200
Impinger #1			
Impinger #2			
Impinger #3			
Impinger #4			

Comments: (Indicate condition of filters, solution colors, presence of precipitates, odor, etc.)

* Filter in good condition; no evidence of leakage. Greyish/black deposit on filter
* Condensate clear
* Impinger solutions clear

FRONT HALF RINSE RESIDUE WEIGHT (Rinse Volume = 212 ml)

G 65.2767 g
T 65.2683 g
N 0.0084 g

Blank correction negligible

Net Residue wt = 0.0096
Net Filter wt = 0.0084

Total Net Particulate Wt = 0.0180 g

Figure A-13. Modified method 5 sample recovery data - Test No. 4 (continued).

Plant/Location Cincinnati Waterworks

Sampling Location Carbon Regenerator Stack

Train Type Mod. Method 5 Run No. Blanks Run Date 8/30/82-9/1/82

Recovery Date _____ Recovered by Kohler/Howes Checked by J. Howes

1.0 CYCLONE CATCH DATA

Cyclone	Sample No.	Tare, g	Gross, g	Net, g
10 μm		N/A		
3 μm				
1 μm				

2.0 FILTER CATCH DATA

Filter Size/Material 3" dia. Pallflex 2500 QAST Quartz Fiber

Filter No.	Sample No.	Tare, g	Gross, g	Net, g
PD-5	BCL-CW-MM5-QPF-Blank-1	0.3711	0.3706	-0.0005
PD-6	BCL-CW-MM5-QPF-Blank-2	0.3686	0.3682	-0.0004

Filter Total _____ g

Figure A-14. Modified method 5 sample recovery data - Blanks.

3.0 CONDENSATE COLLECTION

Total Condensate Collected _____ g

_____ ml

4.0 IMPINGER DATA

Impinger No.	Sample No.	Loading	Initial*	Final*	Gain*
1					
2					
3					
4					
5					

Total Gain* _____

* Indicate whether ml or g

5.0 SILICA GEL WEIGHTS

Initial Weight, g	Final Weight, g	Weight Gain, g

Total Weight Gain _____ g

6.0 TOTAL MOISTURE COLLECTED

Total Collected = Condensate, g + Total Imp. Gain, g + Total Silica Gel Gain, g

= _____ g

7.0 SOLID SORBENT TRAP

Solid Sorbent Material Purified XAD-2

Weight Used 22 g g

Trap No. ST-5

Sample No. BCL-CW-MM5-XAD-Blank

Figure A-14. Modified method 5 sample recovery data - Blanks (continued).

8.0 RINSE SOLUTION DATA

Component	Sample No.	Rinse Composition	Rinse Volume, ml
Front Half	BCL-CW-MM5-FHR-Blank	MeOH/MeCl 50:50	300
Condenser Mod.	BCL-CW-MM5-OMR-Blank	MeCl	~ 200
Impinger #1			
Impinger #2			
Impinger #3			
Impinger #4			

Comments: (Indicate condition of filters, solution colors, presence of precipitates, odor, etc.)

FRONT HALF RINSE RESIDUE (BLANK) Volume = 300 ml

G 64.7262 g
T 64.7284 g
N -0.0022 g

Blank correction negligible

Figure A-14. Modified method 5 sample recovery data - Blanks (continued).

Plant Cincinnati Waterworks

Date 8/30/82

Sampled By J. E. Howes, Jr.

Process Steam ID SL (Scrubber Liquid)

Sampling Location Tap in discharge line

Sampling Method Grab & Composite

Test No. BCL-CW-083082-MM5-1

Composite or Single Sample Composite

Sample Description* Once through effluent from serially connected Venturi and tray scrubbers - Temp. of water ~136 F

Test Started At 1323 hrs

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
1352 hrs	BCL-CW-083082-SL-1-1	16 oz. (~480 ml)	Composited
1423 hrs	" -2	" "	"
1453 hrs	" -3	" "	"
1523 hrs	" -4	" "	"
1553 hrs	" -5	" "	"
1622 hrs	" -6	" "	"
1652 hrs	" -7	" "	"
1722 hrs	" -8	" "	"
	BCL-CW-083082-SL-1-C	128 oz (~1 gal)	Composite of samples
			taken every 1/2 hr.

Figure A-15. Scrubber liquid sampling data - Test No. 1.

Plant Cincinnati Waterworks

Date 8/30/82

Sampled By J. E. Howes, Jr.

Process Steam ID QW

Sampling Location 1/2 Tapline from Quench tank discharge

Sampling Method Grab & Composite

Test No. BCL-CW-083082-MM5-1

Composite or Single Sample Composite

Sample Description* Regenerated carbon quench tank overflow - Temp. 88F
Test started at 1323 hrs

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
1357 hrs	BCL-CW-083082-QW-1-1	16 oz (~480 ml)	Composited
1427 hrs	" -2	" "	"
1455 hrs	" -3	" "	"
1521 hrs	" -4	" "	"
1555 hrs	" -5	" "	"
1624 hrs	" -6	" "	"
1651 hrs	" -7	" "	"
1721 hrs	" -8	" "	"
	BCL-CW-083082-QW-1-C	~128 oz (~1 gal)	Composite of samples taken every 1/2 hr.

Figure A-16. Quench water sampling data - Test No. 1.

Plant Cincinnati Waterworks

Date 8/30/82

Sampled By J. E. Howes, Jr.

Process Steam ID CC (Cyclone Catch)

Sampling Location Hopper discharge drum

Sampling Method Grab & Composite

Test No. BCL-CW-083082-MM5-1

Composite or Single Sample Composite

Sample Description* Fine carbon removed by cyclone from dryer off-gas
Test started at 1323 hrs

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
1352 hrs	BCL-CW-083082-CC-1-1	150 ml (~75 g)	Composited
1423 hrs	" 1-2	" "	"
1453 hrs	" 1-3	" "	"
1523 hrs	" 1-4	" "	"
1553 hrs	" 1-5	" "	"
1623 hrs	" 1-6	" "	"
1652 hrs	" 1-7	" "	"
1722 hrs	" 1-8	" "	"
	BCL-CW-083082-CC-1-C	1200 ml (~600 g)	Composite of samples taken every 1/2 hr.

Figure A-17. Cyclone catch sampling data - Test No. 1.

Plant Cincinnati Waterworks

Date 8/30/82

Sampled By J. E. Howes, Jr.

Process Steam ID SC

Sample Description* Spent carbon feed (wet) to regenerator unit
Test started at 1323 hrs

Sampling Location Drop chute from screw feeder

Sampling Method Grab & Composite

Test No. BCL-CW-083082-MM5-1

Composite or Single Sample Composite

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
1355 hrs	BCL-CW-083082-SC-1-1	150 ml (~130 g)	Composited
1422 hrs	" 1-2	" "	"
1451 hrs	" 1-3	" "	"
1525 hrs	" 1-4	" "	"
1558 hrs	" 1-5	" "	"
1626 hrs	" 1-6	" "	"
1655 hrs	" 1-7	" "	"
1724 hrs	" 1-8	" "	"
	BCL-CW-083082-SC-1-C	1200 ml (~1040 g)	Composite of samples
			taken every 1/2 hrs

Figure A-18. Spent carbon feed sampling data - Test No. 1.

Plant Cincinnati Waterworks

Sampling Location 2" tap in discharge line

Date 8/31/82

Sampling Method Grab & Composite

Sampled By J. E. Howes, Jr.

Test No. BCL-CW-083182-MM5-2

Process Steam ID SL

Composite or Single Sample Composite

Sample Description* Once-through discharge from Venturi and tray scrubber

Temperature ~136 F

Test started at 0847 hrs

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
0916 hrs	BCL-CW-083182-SL-2-1	16 oz (~480 ml)	Composited
0947 hrs	" -2	" "	"
1016 hrs	" -3	" "	"
1047 hrs	" -4	" "	"
1117 hrs	" -5	" "	"
1146 hrs	" -6	" "	"
1217 hrs	" -7	" "	"
1245 hrs	" -8	" "	"
	BCL-CW-083182-SL-2-C	1 gallon	Composite of above samples

Figure A-19. Scrubber liquid sampling data - Test No. 2.

Plant Cincinnati Waterworks

Date 8/31/82

Sampled By J. E. Howes, Jr.

Process Steam ID QW

Sampling Location 1/2" tap in overflow line

Sampling Method Grab & Composite

Test No. BCL-CW-083182-MM5-2

Composite or Single Sample Composite

Sample Description* Overflow water from quench tank. Temperature ~88 F
Test started at 0847 hrs

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
0920 hrs	BCL-CW-083182-QW-2-1	16 oz (~480 ml)	Composited
0946 hrs	" -2	" "	"
1018 hrs	" -3	" "	"
1048 hrs	" -4	" "	"
1119 hrs	" -5	" "	"
1148 hrs	" -6	" "	"
1218 hrs	" -7	" "	"
1247 hrs	" -8	" "	"
	BCL-CW-083182-QW-2-C	1 gallon	Composite of above samples

Figure A-20. Quench water sampling data - Test No. 2.

Plant Cincinnati Waterworks

Date 8/31/82

Sampled By J. E. Howes, Jr.

Process Steam ID CC

Sampling Location Discharge drum

Sampling Method Grab & Composite

Test No. BCL-CW-083182-MM5-2

Composite or Single Sample Composite

Sample Description* Fine carbon removed from dryer off-gas by cyclone

Test started at 0847 hrs

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
0916 hrs	BCL-CW-083182-CC-2-1	150 ml (~75 g)	Composited
0947 hrs	" -2	" "	"
1016 hrs	" -3	" "	"
1047 hrs	" -4	" "	"
1118 hrs	" -5	" "	"
1146 hrs	" -6	" "	"
1217 hrs	" -7	" "	"
1245 hrs	" -8	" "	"
	BCL-CW-083182-CC-2-C	~ 600 g	Composite of above samples

Figure A-21. Cyclone catch sampling data - Test No. 2.

Plant Cincinnati Waterworks

Date 8/31/82

Sampled By J.E. Howes, Jr.

Process Steam ID SC

Sampling Location Drop chute from screw feeder

Sampling Method Grab & Composite

Test No. BCL-CW-083182-MM5-2

Composite or Single Sample Composite

Sample Description* Wet spent carbon feed

Test started at 0847 hrs

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
0922 hrs	BCL-CW-083182-SC-2-1	150 ml (~130 g)	Composited
0949 hrs	" -2	" "	"
1020 hrs	" -3	" "	"
1050 hrs	" -4	" "	"
1121 hrs	" -5	" "	"
1149 hrs	" -6	" "	"
1221 hrs	" -7	" "	"
1248 hrs	" -8	" "	"
	BCL-CW-083182-SC-2-C	~1040 g	Composite of above samples

Figure A-22. Spent carbon feed sampling data - Test No. 2.

Plant Cincinnati Waterworks

Date 8/31/82

Sampled By J. E. Howes, Jr.

Process Steam ID SL (Scrubber Liquid)

Sample Description* Venturi and tray scrubber effluent, Temp ~136 F
Test started at 1443 hrs

Sampling Location 2" tap in discharge line

Sampling Method Grab & Composite

Test No. BCL-CW-083182-MM5-3

Composite or Single Sample Composite

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
1512 hrs	BCL-CW-083182-SL-3-1	16 oz (~480 ml)	Composited
1541 hrs	" -2	" "	"
1612 hrs	" -3	" "	"
1644 hrs	" -4	" "	"
1712 hrs	" -5	" "	"
1743 hrs	" -6	" "	"
1812 hrs	" -7	" "	"
1841 hrs	" -8	" "	"
	BCL-CW-083182-SL-3-C	1 gallon	Composite of above samples

Figure A-23. Scrubber liquid sampling data - Test No. 3.

Plant Cincinnati Waterworks

Date 8/31/82

Sampled By J. E. Howes, Jr.

Process Steam ID QW (Quench water)

Sampling Location 1/2" tap in overflow line

Sampling Method Grab & Composite

Test No. BCL-CW-083182-MM5-3

Composite or Single Sample Composite

Sample Description* Quench tank overflow, Temp ~88F

Test started at 1443 hrs

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
1511 hrs	BCL-CW-083182-QW-3-1	16 oz (~480 ml)	Composited
1542 hrs	" -2	" "	"
1614 hrs	" -3	" "	"
1643 hrs	" -4	" "	"
1714 hrs	" -5	" "	"
1744 hrs	" -6	" "	"
1814 hrs	" -7	" "	"
1843 hrs	" -8	" "	"
	BCL-CW-083182-QW-3-C	1 gallon	Composite of above samples

Figure A-24. Quench water sampling data - Test No. 3.

Plant Cincinnati Waterworks

Date 8/31/82

Sampled By J. E. Howes, Jr.

Process Steam ID CC (Cyclone Catch)

Sampling Location Drop-out drum

Sampling Method Grab & Composite

Test No. BCL-CW-083182-MM5-3

Composite or Single Sample

Sample Description* Fine carbon separated by cyclone from dryer off-gas stream

Test started at 1443 hrs

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
1512 hrs	BCL-CW-083182-CC-3-1	150 ml (~75 g)	Composited
1543 hrs	" -2	" "	"
1612 hrs	" -3	" "	"
1644 hrs	" -4	" "	"
1712 hrs	" -5	" "	"
1742 hrs	" -6	" "	"
1812 hrs	" -7	" "	"
1841 hrs	" -8	" "	"
	BCL-CW-083182-CC-3-C	~ 600 g	Composite of above
			samples

Figure A-25. Cyclone catch sampling data - Test No. 3.

Plant Cincinnati Waterworks

Date 8/31/82

Sampled By J. E. Howes, Jr.

Process Steam ID SC

Sampling Location Drop chute from screw feeder

Sampling Method Grab & Composite

Test No. BCL-CW-083182-MM5-3

Composite or Single Sample Composite

Sample Description* Wet spent carbon feed

Test started at 1443 hrs

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
1515 hrs	BCL-CW-083182-SC-3-1	150 ml (~130 g)	Composited
1545 hrs	" -2	" "	"
1617 hrs	" -3	" "	"
1646 hrs	" -4	" "	"
1721 hrs	" -5	" "	" Feed interrupted to clean eductor
1746 hrs	" -6	" "	"
1816 hrs	" -7	" "	"
1845 hrs	" -8	" "	"
	BCL-CW-083182-SC-3-C	~1040 g	Composite of above samples

Figure A-26. Spent carbon feed sampling data - Test No. 3.

Plant Cincinnati Waterworks

Date 9/1/82

Sampled By J. E. Howes, Jr.

Process Steam ID SL (Scrubber Liquid)

Sample Description Combined effluent from Venturi and tray scrubbers
Temp ~136 F, undissolved solids (carbon)

Sampling Location ~2" tap in discharge line

Sampling Method Grab & Composite

Test No. BCL-CW-090182-MM5-4

Composite or Single Sample Composite

Test started at 0915 hrs

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
0945 hrs	BCL-CW-010182-SL-4-1	16 oz (~480 ml)	Composited
1015 hrs	" -2	" "	"
1045 hrs	" -3	" "	"
1115 hrs	" -4	" "	"
1147 hrs	" -5	" "	"
1215 hrs	" -6	" "	"
1245 hrs	" -7	" "	"
1313 hrs	" -8	" "	"
	BCL-CW-090182-SL-4-C	1 gallon	Composite of above samples

Figure A-27. Scrubber liquid sampling data - Test No. 4.

Plant Cincinnati Waterworks

Date 9/1/82

Sampled By J. E. Howes, Jr.

Process Steam ID QW (Quench Water)

Sample Description* Carbon quench tank overflow water. Temp ~88 F
Test started at 0915 hrs

Sampling Location 1/2" tap in overflow line

Sampling Method Grab & Composite

Test No. BCL-CW-090182-MM5-4

Composite or Single Sample Composite

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
0946 hrs	BCL-CW-090182-OW-4-1	16 oz (~480 ml)	Composited
1016 hrs	" -2	" "	"
1046 hrs	" -3	" "	"
1116 hrs	" -4	" "	"
1148 hrs	" -5	" "	"
1217 hrs	" -6	" "	"
1247 hrs	" -7	" "	"
1315 hrs	" -8	" "	"
	BCL-CW-090182-OW-4-C	1 gallon	Composite of above samples

Figure A-28. Quench water sampling data - Test No. 4.

Plant Cincinnati Waterworks

Sampling Location Drop-out drum

Date 9/1/82

Sampling Method Grab & Composite

Sampled By J. E. Howes, Jr.

Test No. BCL-CW-010182-MM5-4

Process Steam ID CC (Cyclone Catch)

Composite or Single Sample Composite

Sample Description* Fine carbon removed from dryer effluent stream by cyclone

Test started at 0915 hrs

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
0945 hrs	BCL-CW-090182-CC-4-1	150 ml (~75 g)	Composited
1015 hrs	" -2	" "	"
1045 hrs	" -3	" "	"
1115 hrs	" -4	" "	"
1147 hrs	" -5	" "	"
1215 hrs	" -6	" "	"
1245 hrs	" -7	" "	"
1315 hrs	" -8	" "	"
	BCL-CW-090182-CC-4-C	~600 g	Composite of above
			samples

Figure A-29. Cyclone catch sampling data - Test No. 4.

Plant Cincinnati Waterworks

Date 9/1/82

Sampled By J. E. Howes, Jr.

Process Steam ID SC (Spent Carbon)

Sample Description* Spent carbon feed, wet

Test started at 0915 hrs

Sampling Location Drop chute from screw feeder

Sampling Method Grab & Composite

Test No. BCL-CW-090182-MM5-4

Composite or Single Sample Composite

Time Sample Taken	Sample Number	Sample Weight/Volume	Observations
0948 hrs	BCL-CW-090182-SC-4-1	150 ml (~130 g)	Composited
1017 hrs	" -2	" "	"
1048 hrs	" -3	" "	"
1118 hrs	" -4	" "	"
1151 hrs	" -5	" "	"
1219 hrs	" -6	" "	"
1248 hrs	" -7	" "	"
1317 hrs	" -8	" "	"
	BCL-CW-090182-SC-4-C	~ 1040 g	Composite of above samples

Figure A-30. Spent carbon feed sampling data - Test No. 4.

Plant/Address: Cincinnati Waterworks
Date: 8/31/82
Sampling Location: Tap off N.G. supply to boiler
Source Code: CW
Run Number: BCI-CW-083182-PCB-1
Operator: Howes
Ambient Temperature, °F: 82
Barometric Pressure, in. Hg: 29.47 in. Hg
Static Pressure, in. Hg: N/A

Pre Test Leak Rate: <0.001 cfm @ -15 in. Hg
Post Test Leak Rate: <0.001 cfm @ -5 in. Hg

Probe Length and Type: N/A S/N:
Nozzle S/N: Nozzle ID: N/A
Assumed Moisture, %: N/A
XAD 2 Module S/N: PCB-1
Meter Box S/N: 546-1 Oven S/N: N/A
Meter $\Delta H_@$: N/A
C Factor: N/A
Probe Heater Setting: N/A
Oven Setting: N/A
Reference ΔP: N/A
Calculations Checked By: Howes Date:

Traverse Point Number	Sampling Time, min	Clock Time (24 hr Clock)	Gas Meter Reading, ft^3	Velocity Head, in. H_2O	Orifice Pressure Differential, in. H_2O Desired	Orifice Pressure Differential, in. H_2O Actual	Stack Temperature, °F	Dry Gas Meter Temperature Inlet, °F	Dry Gas Meter Temperature Outlet, °F	Pump Vacuum, in. Hg	Oven Temperature, °F	Impinger Temperature, °F	Sorbent Trap Temperature, °F	Probe Temperature, °F
	0	1445	49.7744						81	-0			82	
	15	1500	55.35						81	-0			82	
	30	1515	61.55						81	-0			82	
	46	1531	68.39						81	-0			82	
	60	1545	74.39						81	-0			82	
	75	1600	81.28						81	-0			82	
	90	1615	87.69						81	-0			82	
	105	1630	94.28						82	-0			83	
	120	1645	101.2848						82	-0			83	
				$V_m = (17.64)(51.5104)\left(\frac{29.47}{81.3 + 460}\right) = 49.469$ scf										
									= 1.40 scm					
Total	120		51.5104											
Average								81.3						

Figure A-31. Natural gas supply PCB sampling data

APPENDIX B

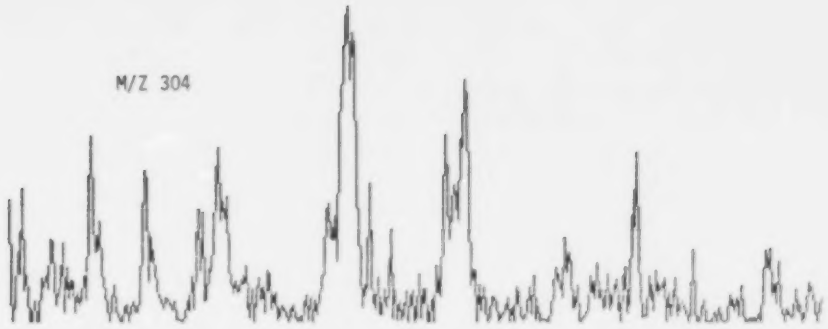
MASS SPECTRA DATA

Typical HRGC/HRMS selected ion current profiles for TCDDs, TCDFs, and the 2,3,7,8-TCDD-$^{13}C_{12}$ internal standards are shown in Figures B-1 through B-18. Figures B-19 through B-21 show typical selected ion current profiles of the 2,3,7,8-TCDD/2,3,7,8-TCDD-$^{13}C_{12}$ standard used to check the HRGC/HRMS calibration during analyses of the samples. Profiles of the tri- and tetrachlorobiphenyls found in the natural gas supply are presented in Figures B-22 through B-26. In all TCDD, TCDF, and PCB profiles, the most intense peak has been normalized to full scale. The following exact masses were monitored for acquisition of the TCDD and TCDF selected ion current profiles.

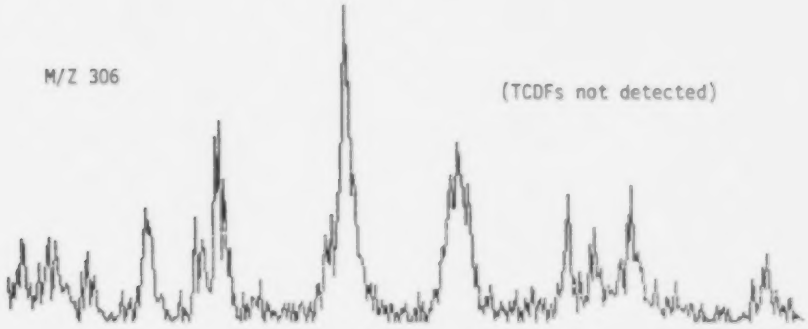
TCDF isomers	303.9016
TCDF isomers	305.8987
TCDD isomers	319.8965
TCDD isomers	321.8936
2,3,7,8-TCDD-$^{13}C_{12}$ internal standard	331.9368
2,3,7,8-TCDD-$^{13}C_{12}$ internal standard	333.9338

SIR REPORT. 097430.SD RUN - 1 2.OUL, 37901-51-13, C.W.

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUND
0 HRS 16 MINS 2 SECS			0.0000	0.0000		0.0000



0 HRS 16 MINS 2 SECS 0.0000 0.0000 0.0000 0.

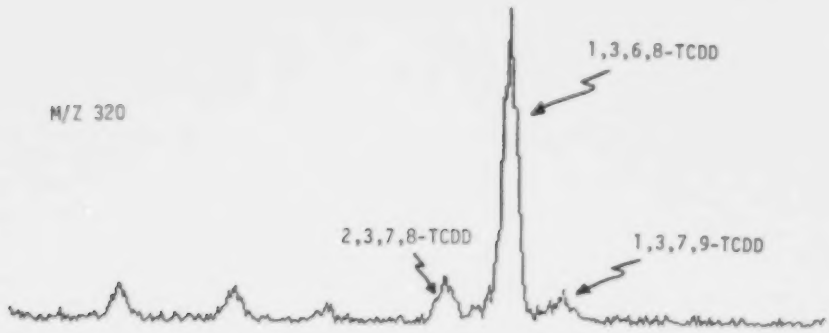


Test No. 2 - Mod. Method 5 Particulate Sample
Column - 60m SP2340 Fused Silica

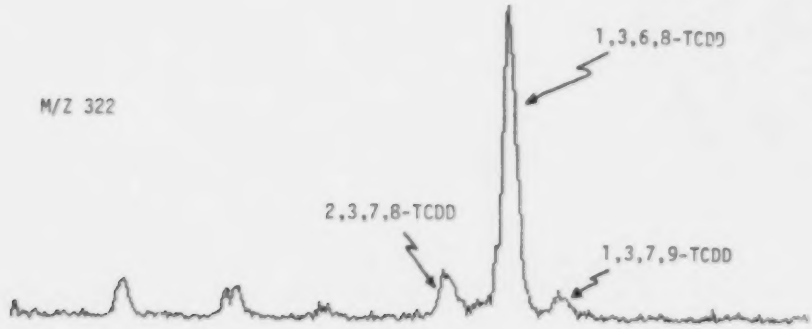
Figure B-1. HRGC/HRMS selected ion current profile for TCDFs.

SIR REPORT. 097430.SD RUN - 1 2.OUL. 37901-51-13, C.W.

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUND
0 HRS 16 MINS 1 SECS			0.0000	0.0000		0.0000



0 HRS 0 MINS 0 SECS			0.0000	0.0000	0.0000	0.

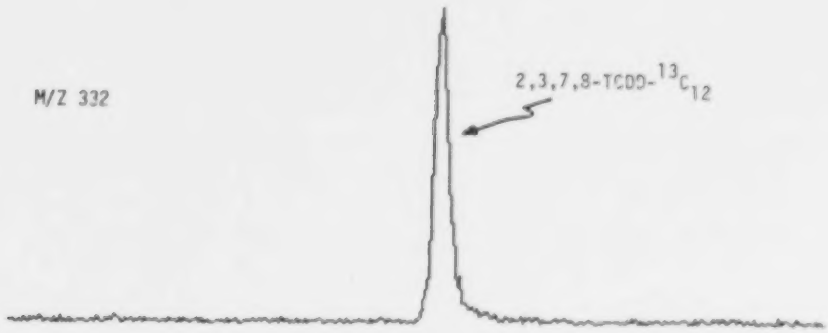


Test No. 2 - Mod. Method 5 Particulate Sample
Column - 60m SP2340 Fused Silica

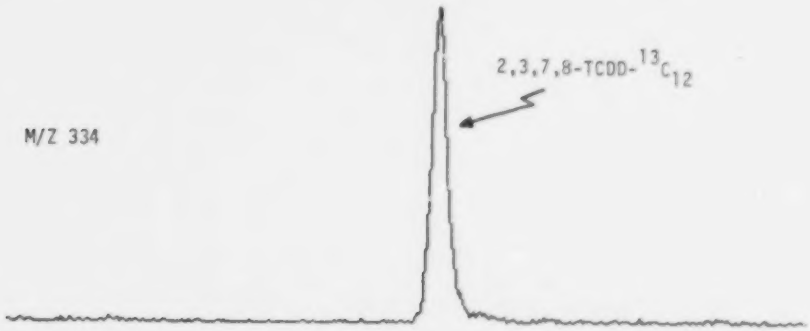
Figure B-2. HRGC/HRMS selected ion current profile for TCDDs.

SIR REPORT. 097430.SD RUN - 1 2.0UL, 37901-51-13, C.W.

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUND
0 HRS 0 MINS 0 SECS			0.0000	0.0000		0.0000



0 HRS 0 MINS 0 SECS			0.0000	0.0000	0.0000	0.

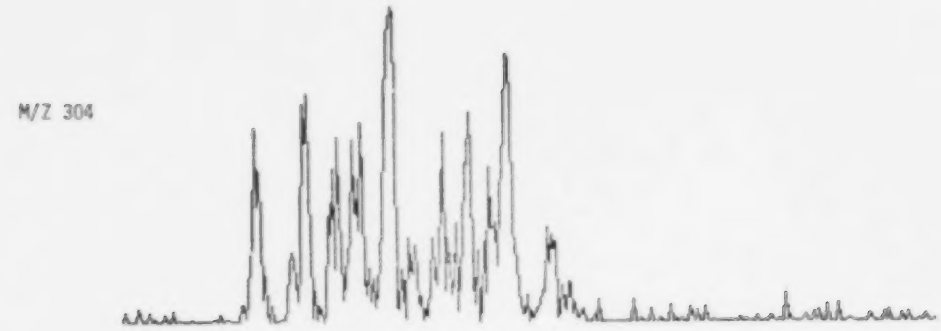


Test No. 2 - Mod. Method 5 Particulate Sample
Column - 60m SP2340 Fused Silica

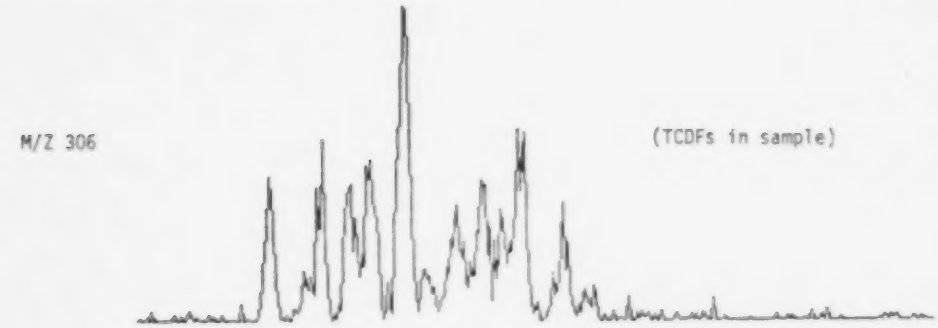
Figure B-3. HRGC/HRMS selected ion current profile for internal standard.

SIR REPORT. 096015.SD RUN - 1 2.OUL, 37901-5-8, C.W.

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUND
0 HRS	0 MINS	0 SECS	0.0000	0.0000		0.0000



0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000 0.

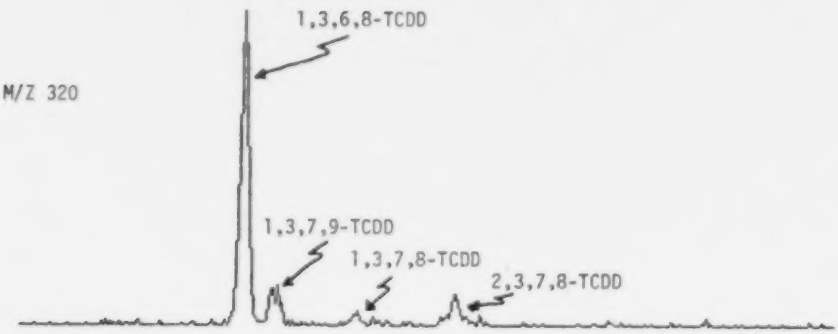


Test No. 2 - Mod. Method 5 XAD-2 Trap
Column - 30m DB-5 Fused Silica

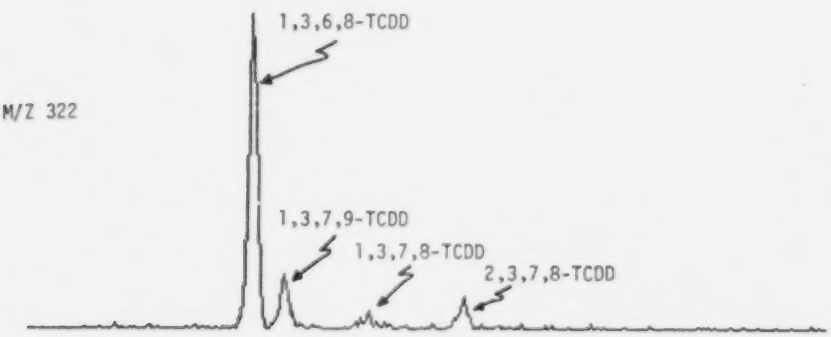
Figure B-4. HRGC/HRMS selected ion current profile for TCDFs.

SIR REPORT. 096015.SD RUN - 1 2.0UL, 37901-5-8, C.W.

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUND
0 HRS	0 MINS	0 SECS	0.0000	0.0000		0.0000



0 HRS	0 MINS	0 SECS	0.0000	0.0000	0.0000	0.

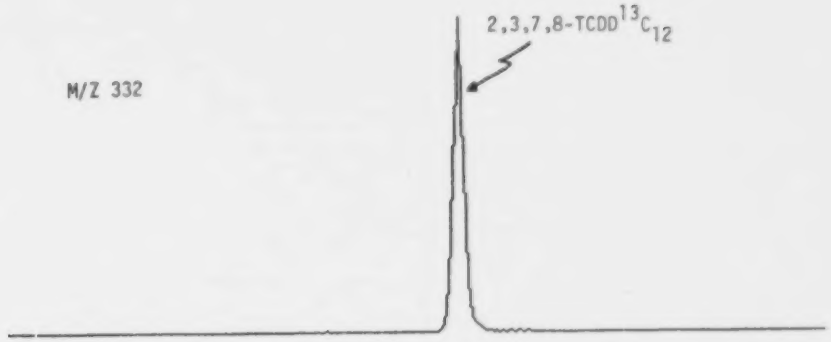


Test No. 2 - Mod. Method 5 XAD-2 Trap
Column - 30m DB-5 Fused Silica

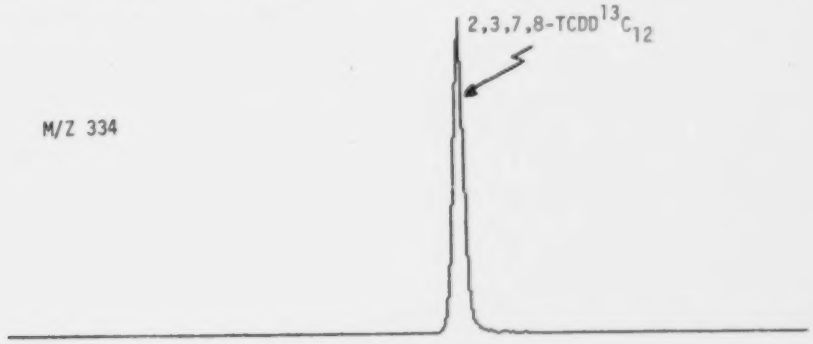
Figure B-5. HRGC/HRMS selected ion current profile for TCDDs.

SIR REPORT. 096015.SD RUN - 1 2.0UL. 37901-5-8. C.W.

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUND
0 HRS 0 MINS 0 SECS			0.0000	0.0000		0.0000



0 HRS 11 MINS 32 SECS 0.0000 0.0000 0.0000 0.

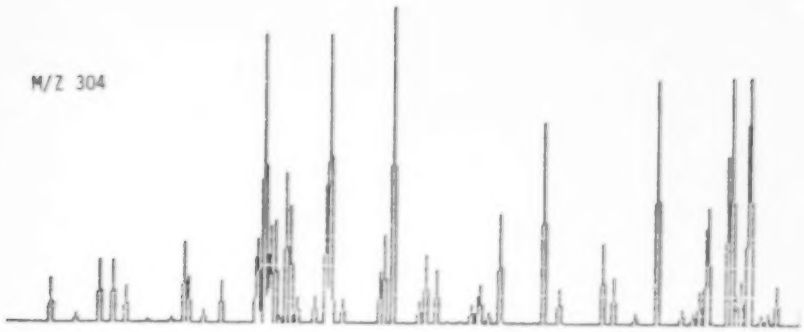


Test No. 2 - Mod. Method 5 XAD-2 Trap
Column - 30m DB-5 Fused Silica

Figure B-6. HRGC/HRMS selected ion current profile for internal standard.

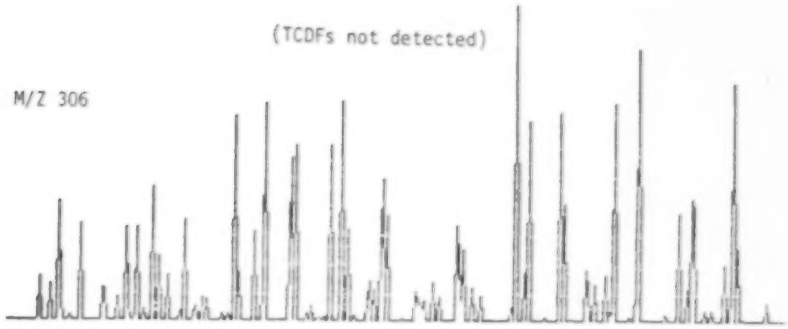
SIR REPORT. 096124.SD RUN - 1 2.0UL, 37901-12-20, C.W. RUN 2 SL

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUNI
0 HRS	0 MINS	0 SECS	0.0000	0.0000		0.0000



0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000

(TCDFs not detected)

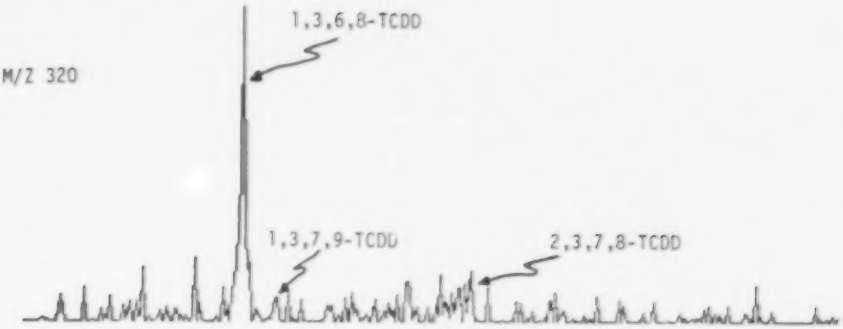


Test No. 2 - Scrubber Liquid
Column - 30m DB-5 Fused Silica

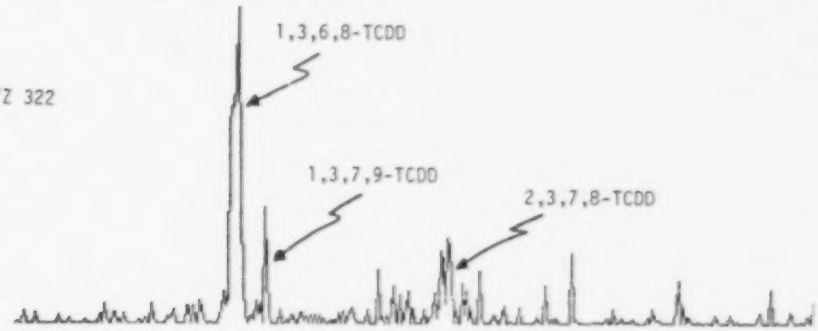
Figure B-7. HRGC/HRMS selected ion current profile for TCDFs.

SIR REPORT. 096124.SD RUN - 1 2.0UL, 37901-12-20, C.W. RUN 2 SL

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUND
0 HRS	0 MINS	0 SECS	0.0000	0.0000		0.0000



0 HRS	0 MINS	0 SECS	0.0000	0.0000	0.0000	0.

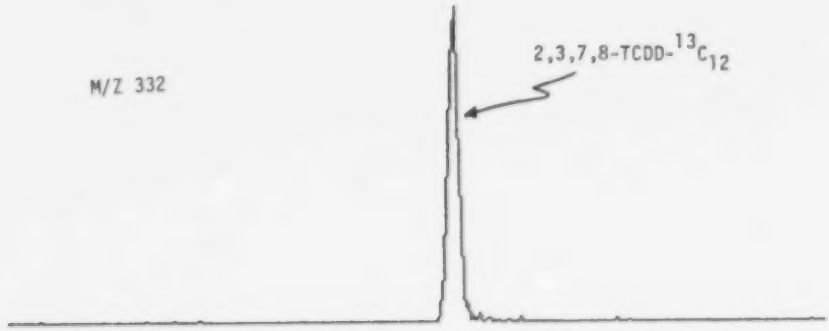


Test No. 2 - Scrubber Liquid
Column - 30m DB-5 Fused Silica

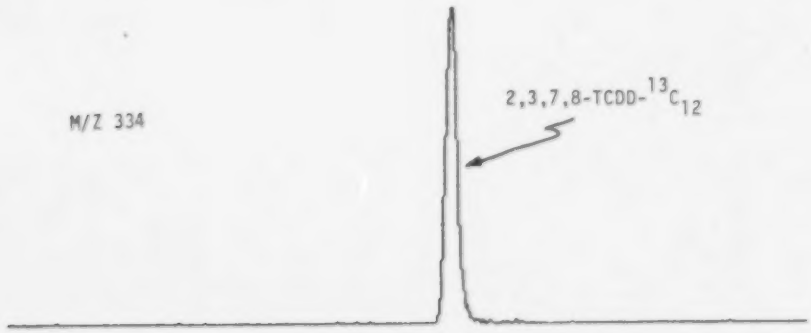
Figure B-8. HRGC/HRMS selected ion current profile for TCDDs.

SIR REPORT. 096124.SD RUN - 1 2.OUL, 37901-12-20, C.W. RUN 2 SL

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUND
0 HRS 0 MINS 0 SECS			0.0000	0.0000		0.0000



0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000 0.

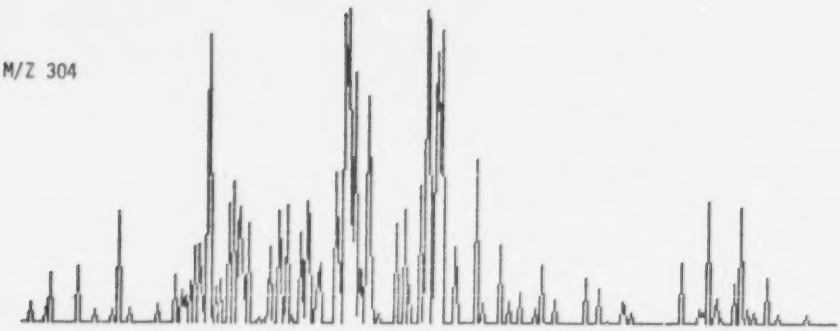


Test No. 2 - Scrubber Liquid
Column - 30m DB-5 Fused Silica

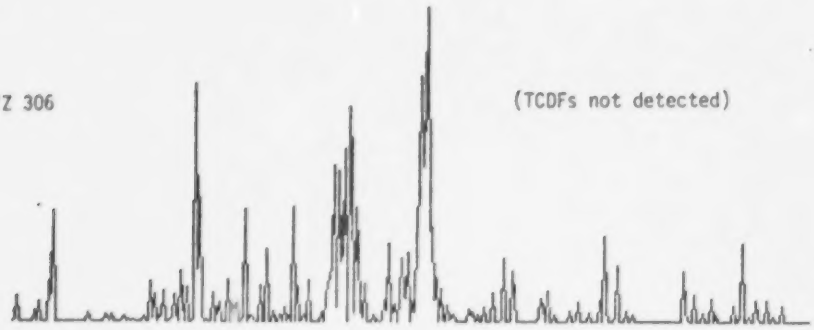
Figure B-9. HRGC/HRMS selected ion current profile for internal standard.

SIR REPORT. 096119.SD RUN - 1 2.OUL, 37901-12-22, C.W. RUN 2 QW

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUND
0 HRS 0 MINS 0 SECS			0.0000	0.0000		0.0000



0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000 0.

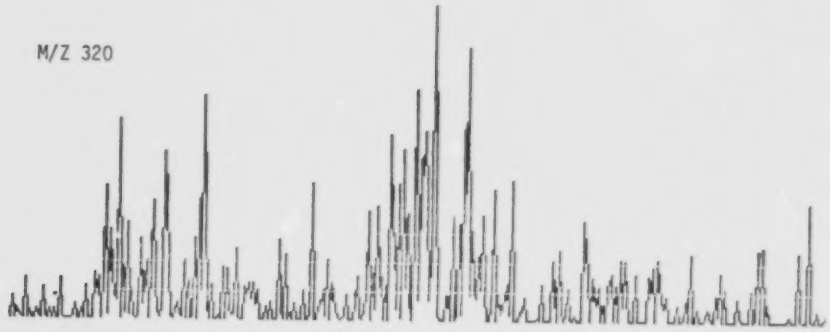


Test No. 2 - Quench Water
Column - 30m DB-5 Fused Silica

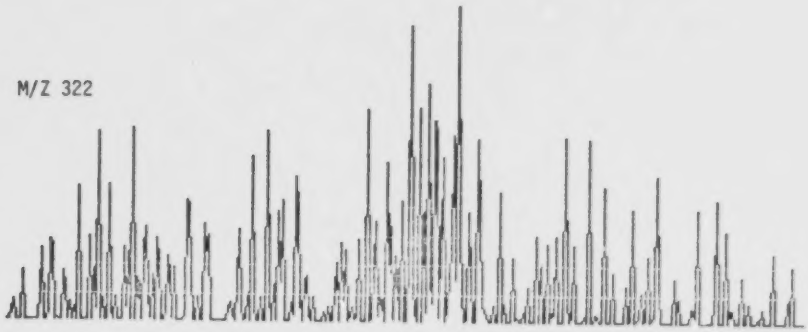
Figure B-10. HRGC/HRMS selected ion current profile for TCDFs.

SIR REPORT. 096119.SD RUN - 1 2.OUL, 37901-12-22, C.W. RUN 2 GW

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUND N
0 HRS 0 MINS 0 SECS			0.0000	0.0000		0.0000



0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000 0.

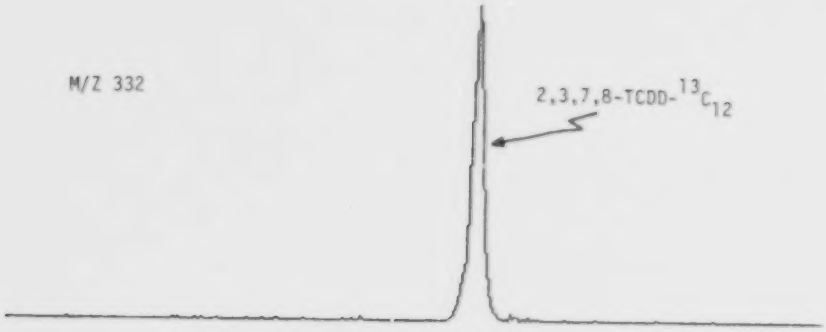


Test No. 2 - Quench Water
Column - 30m DB-5 Fused Silica

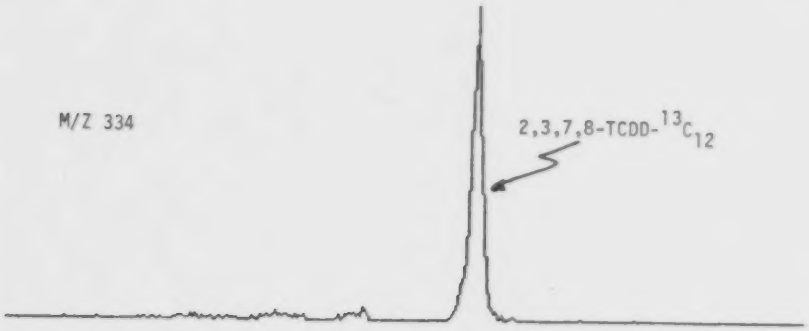
Figure B-11. HRGC/HRMS selected ion current profile for TCDDs.

SIR REPORT. 096119.SD RUN - 1 2.OUL. 37901-12-22. C.W. RUN 2 QW

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUND
0 HRS 0 MINS 0 SECS			0.0000	0.0000		0.0000



0 HRS 0 MINS 0 SECS			0.0000	0.0000	0.0000	0.



Test No. 2 - Quench Water
Column - 30m DB-5 Fused Silica

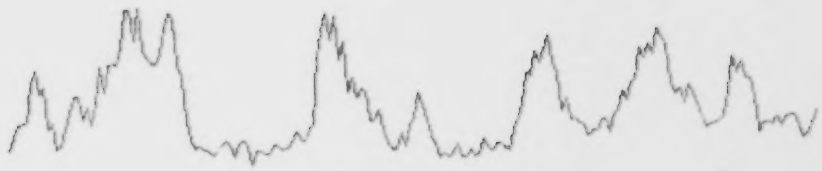
Figure B-12. HRGC/HRMS selected ion current profile for internal standard.

SIR REPORT. FILE - 097124 .SD RUN - 1 2.OUL. 37901-49-13. C.W

UNCALIBRATED.

0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000

M/Z 304



0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000

M/Z 306

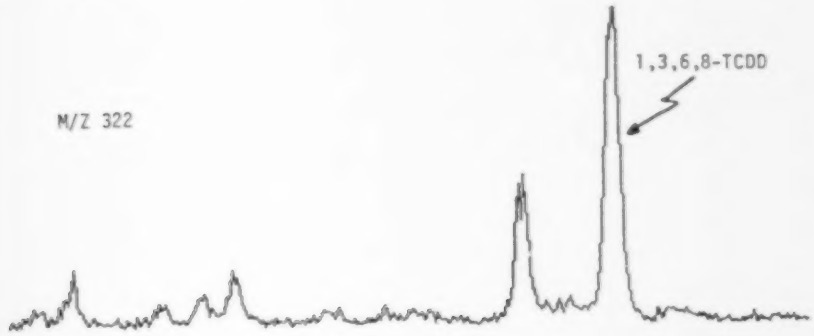
Test No. 2- Cyclone Catch
Column - 60m SP2340 Fused Silica

Figure B-13. HRGC/HRMS selected ion current profile for TCDFs.

SIR REPORT. 097124.SD RUN - 1 2.0UL, 37901-49-13, C.W.

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUND
0 HRS 16 MINS 1 SECS			0.0000	0.0000		0.0000

0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000 0.

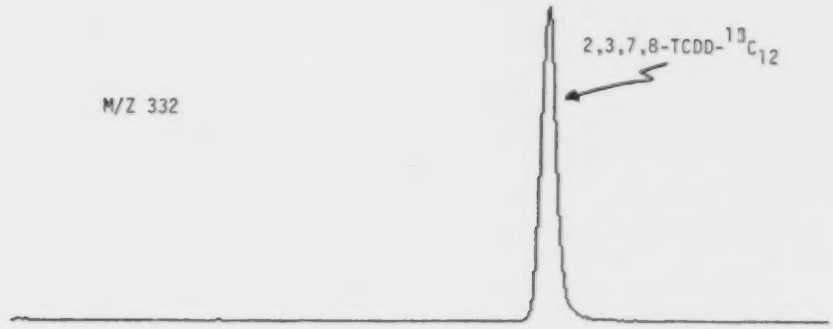


Test No. 2 - Cyclone Catch
Column - 60m SP2340 Fused Silica

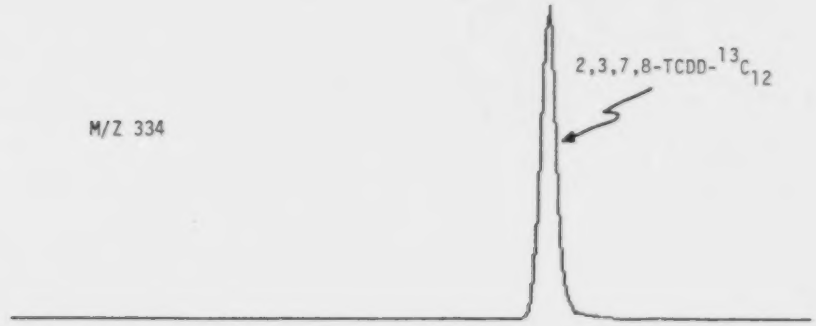
Figure B-14. HRGC/HRMS selected ion current profile for TCDDs.

SIR REPORT. 097124.SD RUN - 1 2.OUL. 37901-49-13. C.W.

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUND
0 HRS 0 MINS 0 SECS			0.0000	0.0000		0.0000



0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000 0.

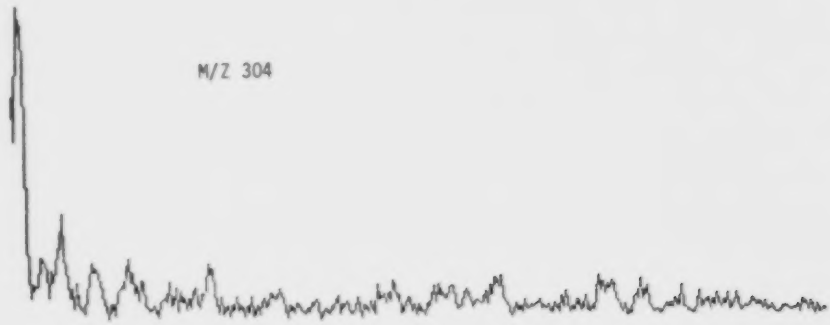


Test No. 2 - Cyclone Catch
Column - 60m SP2340 Fused Silica

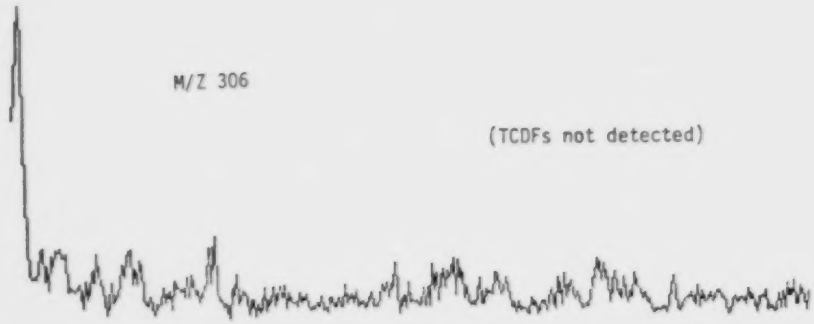
Figure B-15. HRGC/HRMS selected ion current profile for internal standard.

SIR REPORT. 096214.SD RUN - 1 2.OUL, 37901-26-14, C.W.

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUND
0 HRS	0 MINS	0 SECS	0.0000	0.0000		0.0000



0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000 0.

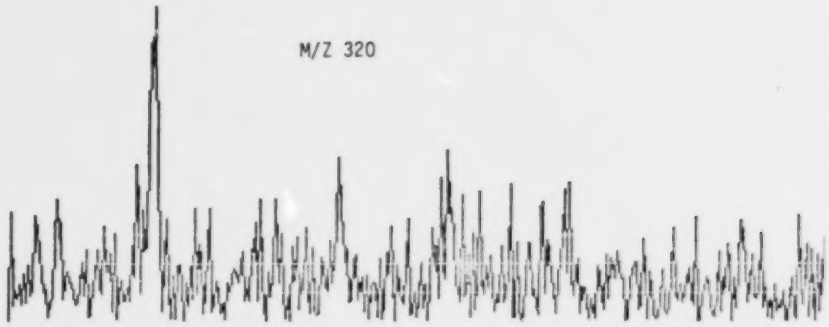


Test No. 2 - Spent Carbon Feed
Column - 30m DB-5 Fused Silica

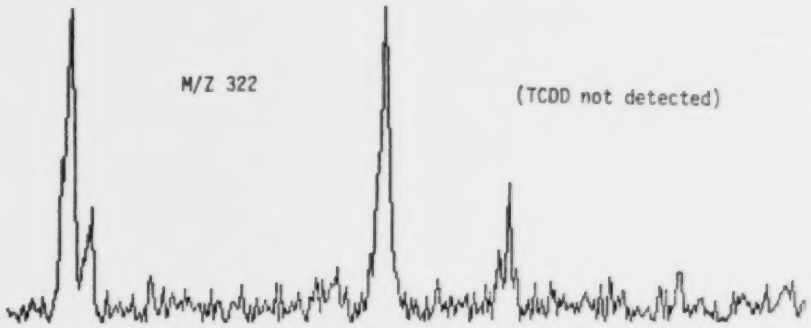
Figure B-16. HRGC/HRMS selected ion current profile for TCDDs.

SIR REPORT. 096214.SD RUN - 1 2.OUL, 37901-26-14, C.W.

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUND
0 HRS 11 MINS 1 SECS			0.0000	0.0000		0.0000



0 HRS 0 MINS 0 SECS 0.0000 0.0000 0.0000 0.

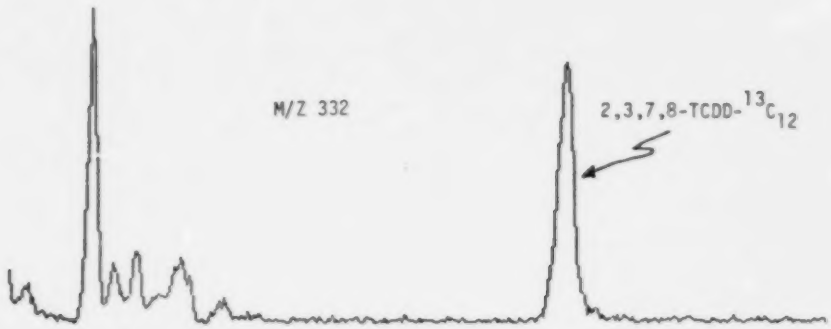


Test No. 2 - Spent Carbon Feed
Column - 30m DB-5 Fused Silica

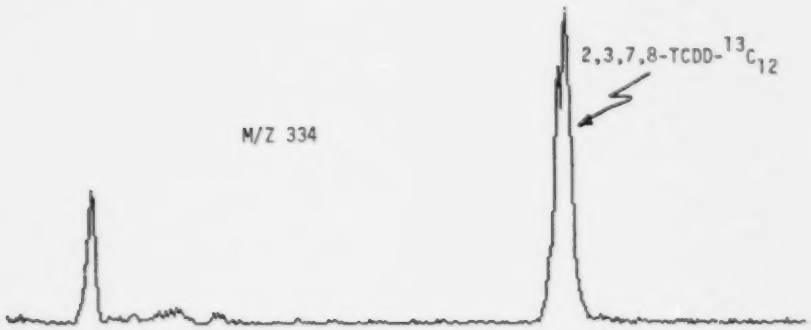
Figure B-17. HRGC/HRMS selected ion current profile for TCDDs.

SIR REPORT. 096214.SD RUN - 1 2.0UL, 37901-26-14, C.W.

RETN. TIME	MASS	HEIGHT	AREA	CONC. (H)	CONC. (A)	COMPOUND
0 HRS 11 MINS 2 SECS			0.0000	0.0000		0.0000



0 HRS 11 MINS 2 SECS	0.0000	0.0000	0.0000	0.

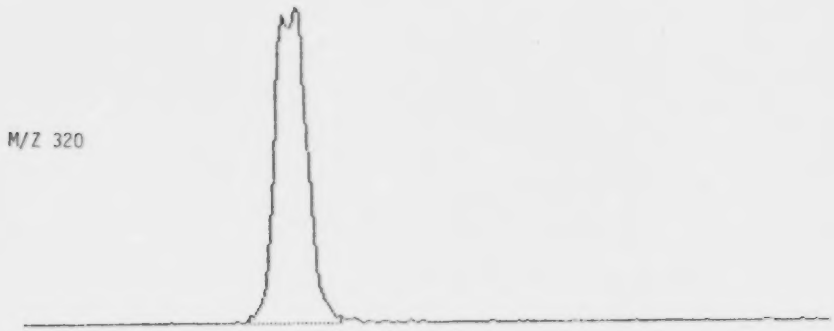


Test No. 2 - Spent Carbon Feed
Column - 30m DB-5 Fused Silica

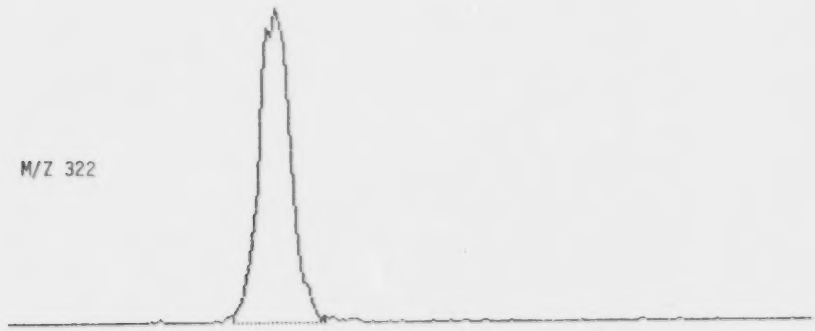
Figure B-18. HRGC/HRMS selected ion current profile for internal standard.

SIR REPORT. FILE - 096318 .SD RUN - 1 2.0UL, 20/95PG/UL N/C-13 TCI

0 HRS 13 MINS 42 SECS 152.9095 661.0274 0.0000



0 HRS 13 MINS 41 SECS 214.7046 903.0380 0.0000

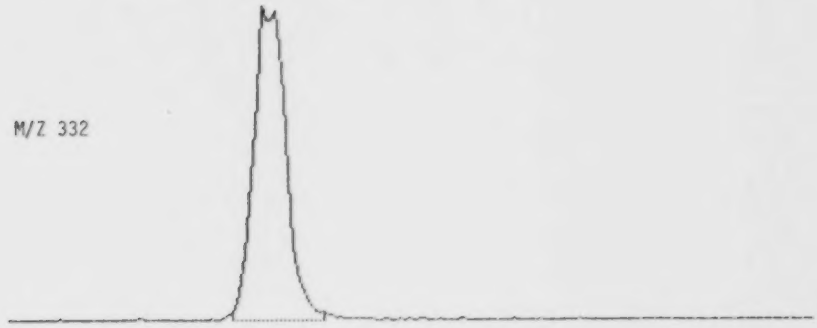


Standard - 0.04 ng 2,3,7,8-TCDD/0.19 ng 2,3,7,8-TCDD-$^{13}C_{12}$
Column - 60 m SP 2340 Fused Silica

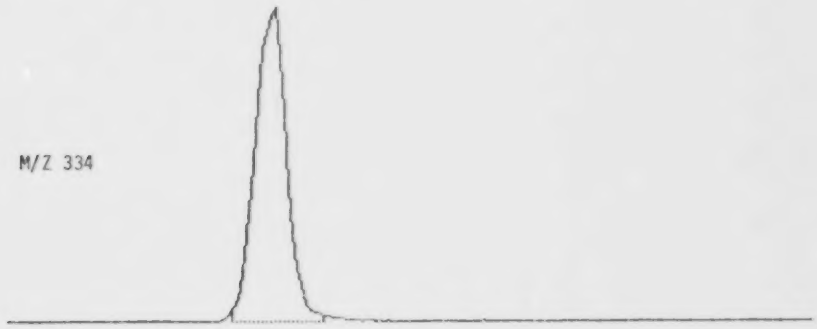
Figure B-19. Typical HRGC/HRMS selected ion current profile for 2,3,7,8-TCDD (m/z 320 and m/z 322)

SIR REPORT. FILE - 096318 .SD RUN - 1 2.0UL, 20/95PG/UL N/C-13 TCI

0 HRS 13 MINS 40 SECS 755.0548 3296.8340 0.0000



0 HRS 13 MINS 42 SECS 910.2708 3845.1030 0.0000

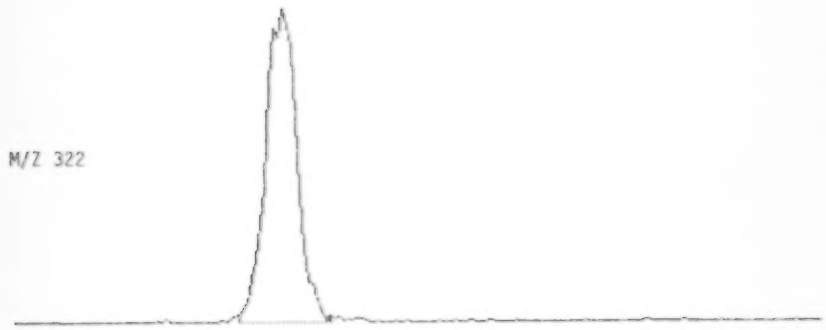


Standard - 0.04 ng 2,3,7,8-TCDD/0.19 ng 2,3,7,8-TCDD-$^{13}C_{12}$
Column - 60 m SP 2340 Fused Silica

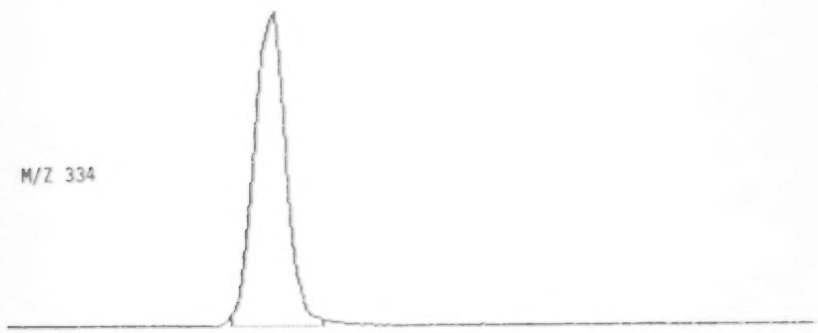
Figure B-20. Typical HRGC/HRMS selection ion current profile for 2,3,7,8-TCDD-$^{13}C_{12}$ (m/z 332 and m/z 334)

SIR REPORT FILE - 096318 SD RUN - 1 2.0UL. 20/95PG/UL N/C-13 TCI

0 HRS 13 MINS 41 SECS 214.7046 903.0380 0.0000



0 HRS 13 MINS 42 SECS 910.2708 3845.1030 0.0000



Standard - 0.04 ng 2,3,7,8-TCDD/0.19 ng 2,3,7,8-TCDD-$^{13}C_{12}$
Column - 60 m SP2340 Fused Silica

Figure B-21. Typical HRGC/HRMS selected ion current profile for 2,3,7,8-TCDD (m/z 322) and 2,3,7,8-TCDD-$^{13}C_{12}$ (m/z 334)

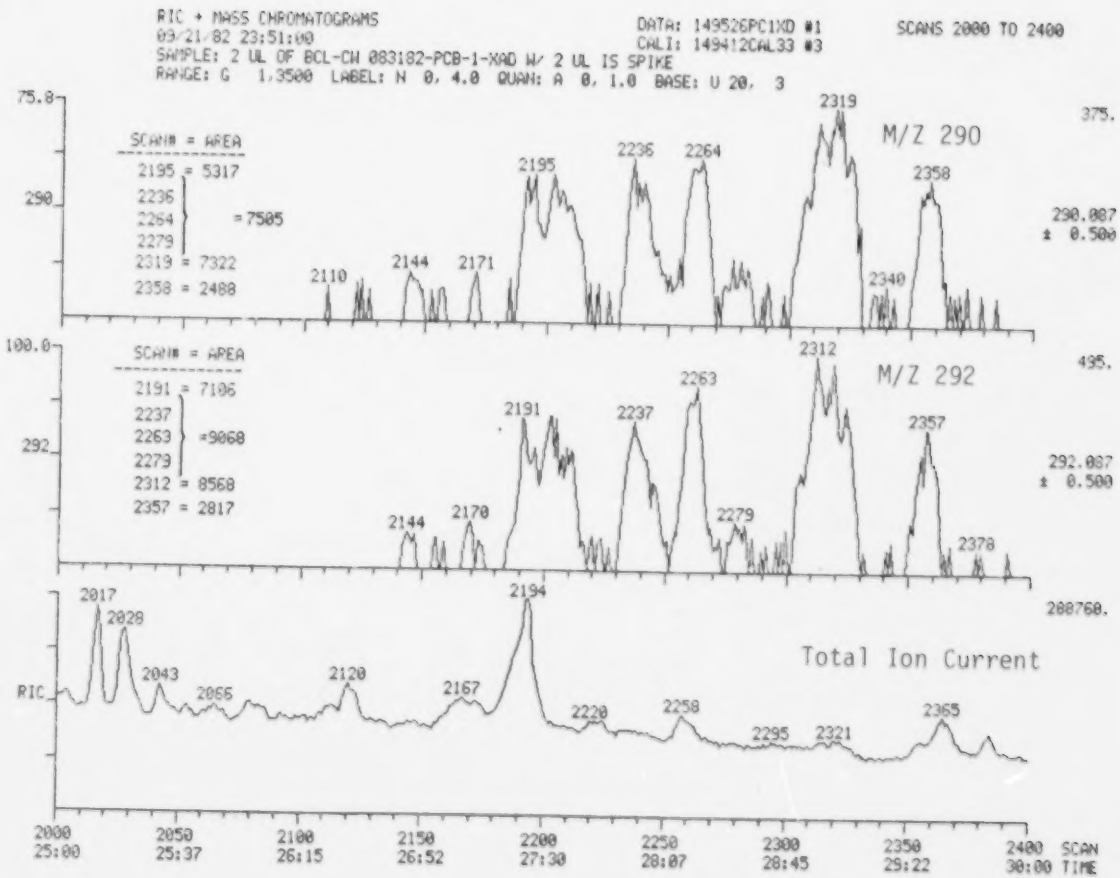


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Figure B-22. Reconstructed ion chromatogram for tetrachlorobiphenyl region.

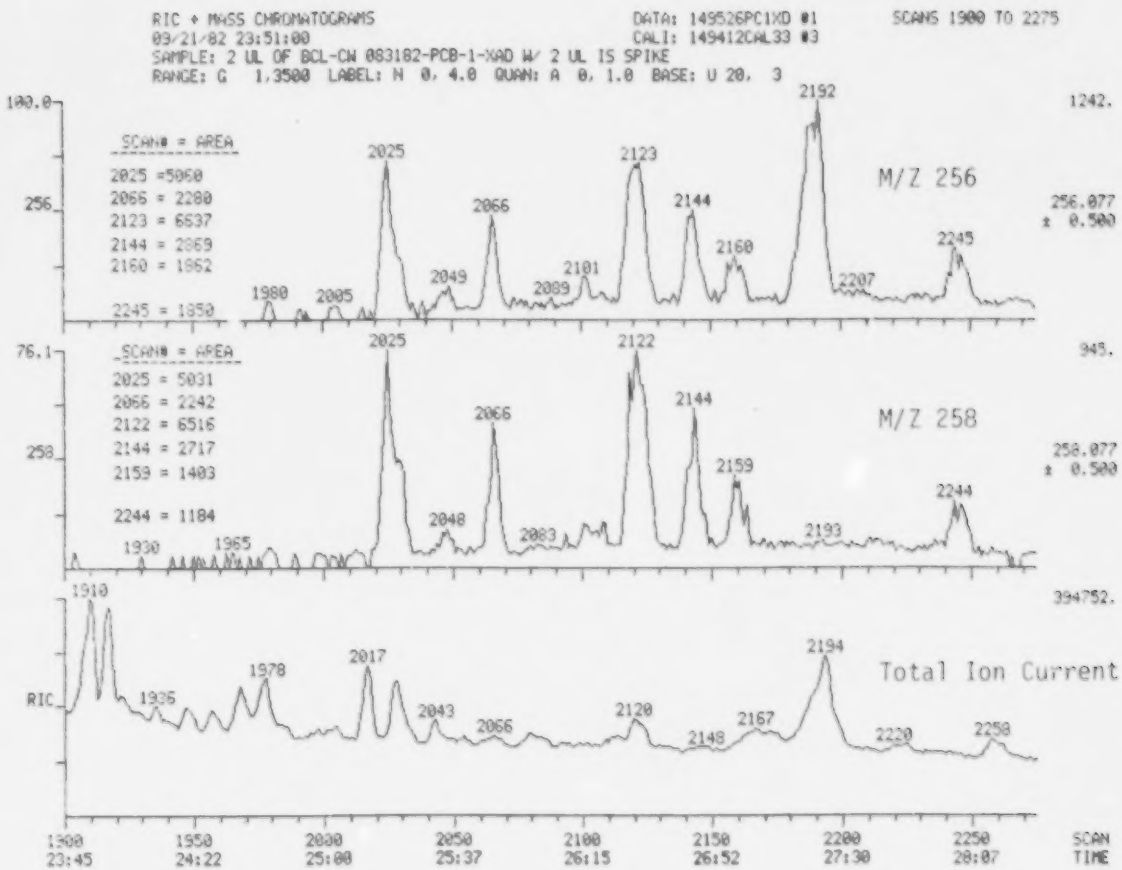


Figure B-23. Reconstructed ion chromatograms for trichlorobiphenyl region.

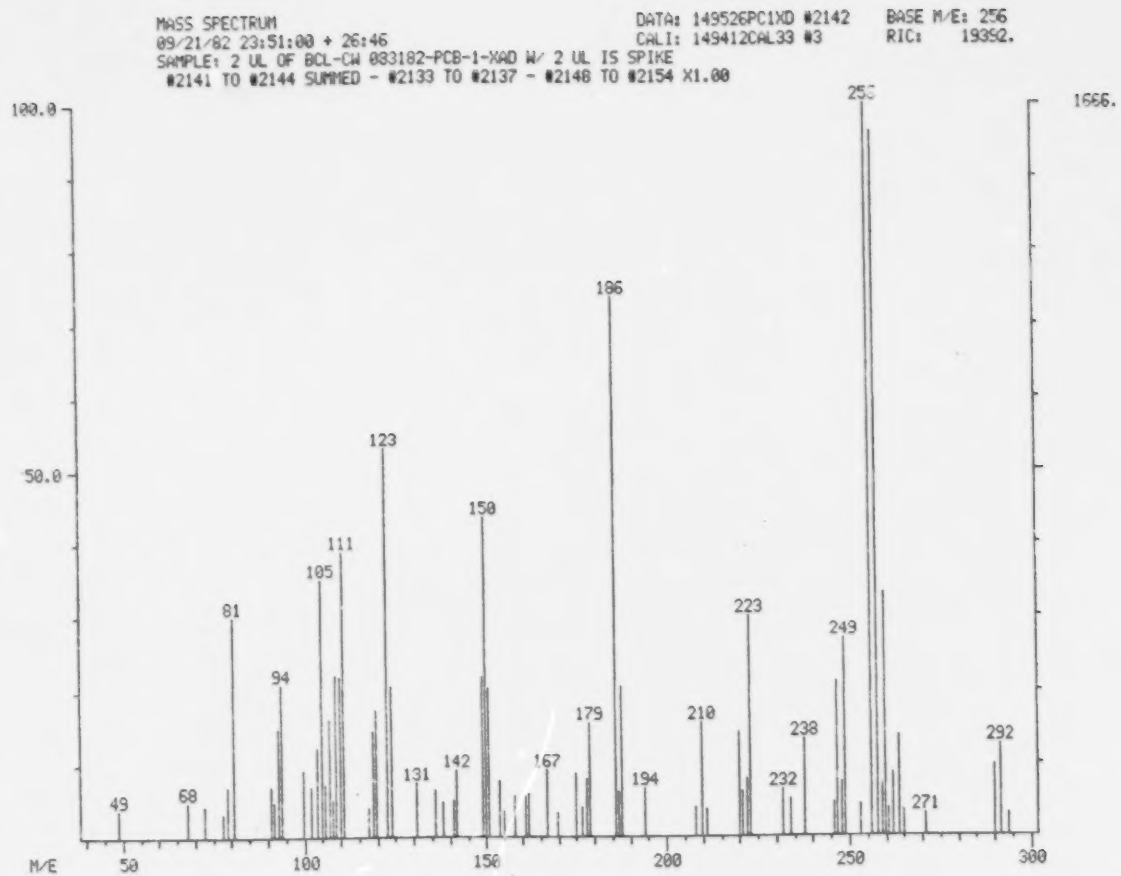


Figure B-24. Typical mass spectrum for trichlorobiphenyls (Scan No. 2142).

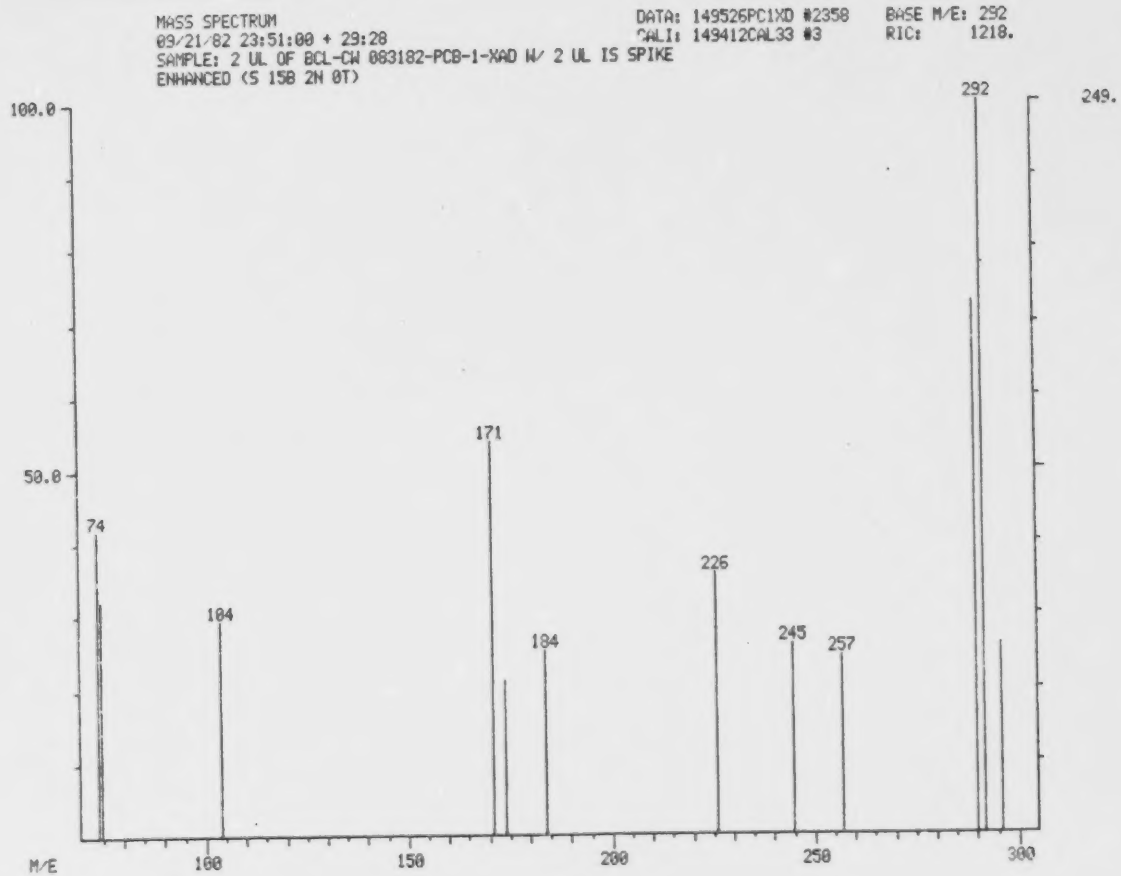


Figure B-25. Typical mass spectrum for tetrachlorobiphenyls (Scan No. 2358).

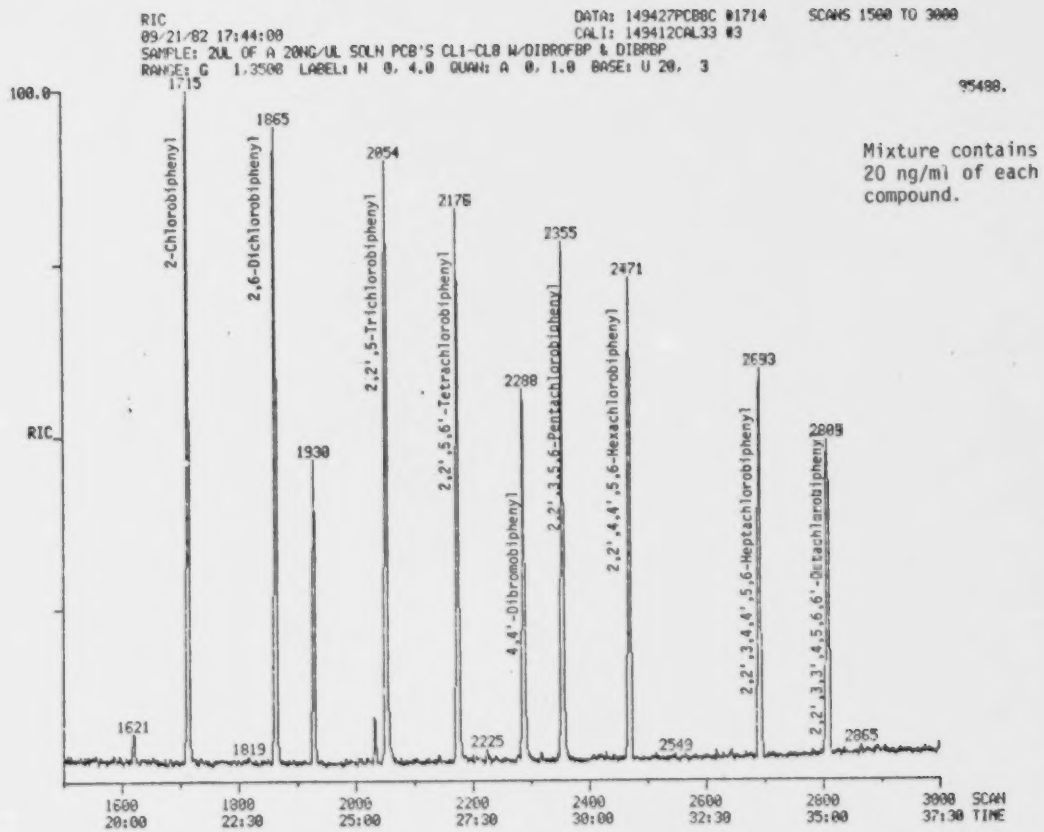


Figure B-26. Reconstructed ion chromatogram of PCB standard mixture.

APPENDIX C

CALIBRATION DATA

Calibration data for components of the modified Method 5 train used to sample the reactivator stack emissions are given in Figures C-1 through C-6. Figure C-7 presents the calibration data for the dry gas meter used to sample the natural gas supply for PCBs.

Calibration Date 8/26/82

Performed by D. F. Kohler

Barometer (P_b) 29.38 in. Hg

Sample Box S/N 1971

Dry Gas Meter S/N

Wet Test Meter S/N Sec. 569

Orifice Manometer, in. H_2O (ΔH)	Gas Volumes, ft^3		Temperatures, F				Time, min. θ	Ratio V_w/V_d, γ	Dry Test $\Delta H@$, in. H_2O
	Wet Test (V_w)	Dry Gas (V_d)	Wet Test (t_w)	Dry Gas Inlet	Dry Gas Outlet	Dry Gas Avg. (t_d)			
0.5	5	I 830.000 / F 834.785	75	70/81	72/76	74.75	12.35	1.043	1.76
1.0	5	I 834.785 / F 839.627	75	79/91	76/80	81.50	8.95	1.042	1.83
2.0	10	I 839.627 / F 849.436	75	88/98	80/87	88.25	13.325	1.039	2.00
4.0	10	I 849.436 / F 859.356	75	95/104	86/90	93.75	9.583	1.033	2.05
		I / F							
		I / F							
							Average	1.039	1.91

Figure C-1. Method 5 dry gas meter pre-sampling calibration data.

Calibration Date 9/7/82 Sample Box S/N 1971

Performed by Kohler Dry Gas Meter S/N

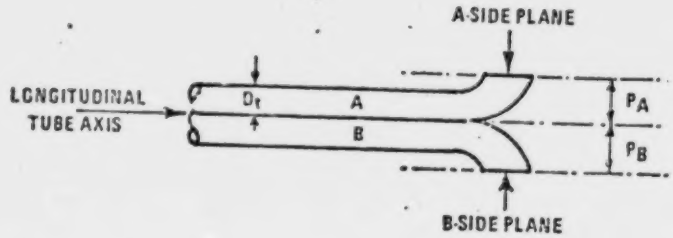
Barometer (P_b) 29.48 in. Hg Wet Test Meter S/N #569

Orifice Manometer, in. H_2O (ΔH)	Gas Volumes, ft^3		Temperatures, F				Time, min. θ	Ratio V_w/V_d, γ	Dry Test $\Delta H@$, in. H_2O
	Wet Test (V_w)	Dry Gas (V_d)	Wet Test (t_w)	Dry Gas Inlet	Dry Gas Outlet	Dry Gas Avg. (t_d)			
0.5	5	I 590.370 / F 595.154	78.8	72/83	74/78	76.8	12.166	1.040	1.72
1.0	5	I 595.154 / F 600.001	78.8	83/92	78/83	84	8.77	1.039	1.77
2.0	10	I 600.001 / F 609.851	78.8	91/103	83/89	91.5	13.33	1.034	2.01
4.0	10	I 609.851 / F 619.750	78.8	99/112	89/93	98.3	9.533	1.036	2.03
		I / F							
		I / F							
							Average	1.037	1.88

Figure C-2. Method 5 dry gas meter post-sampling calibration data.

Pitot Tube S/N 549-44-1 Performed By J. Howes Date 8/26/82

1.0 PITOT TUBE DIMENSIONS

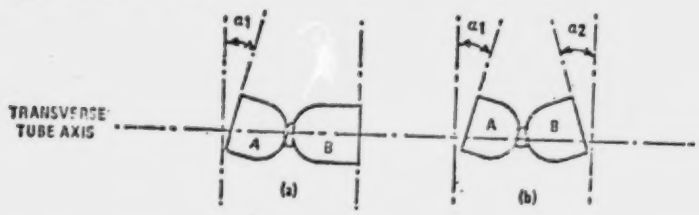


D_t = 0.95 cm (D_t must be between 0.48 and 0.95 cm)

P_A = 1.4 cm (P_A and P_B must be equal)

P_B = 1.4 cm (P_A and P_B must be between 1.05 and 1.50 D_t)

2.0 PITOT TUBE ASSEMBLY

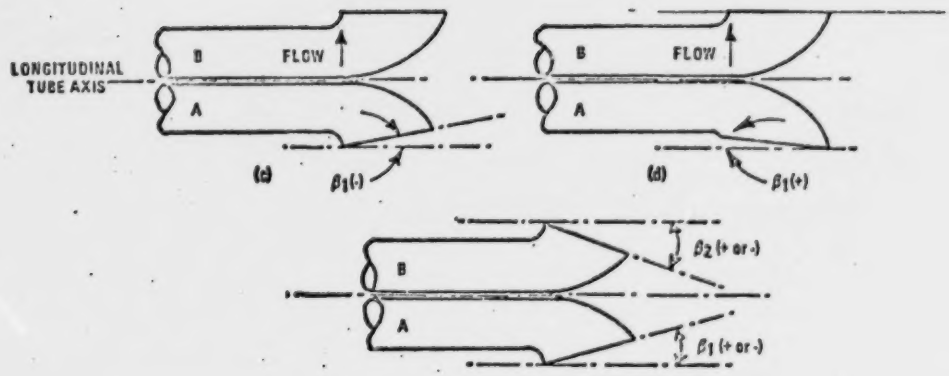


α_1 = <5 degrees

α_2 = <5 degrees

(α_1 and α_2 must be < 10°)

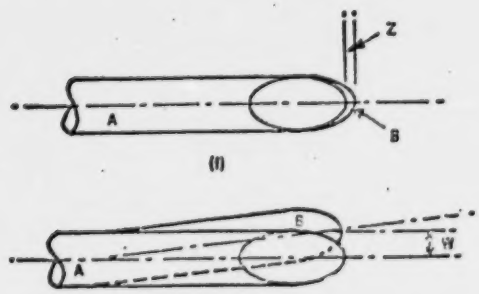
Figure C-3. Method 5 pitot tube calibration data.



B_1 = <3 degrees

(B_1 and B_2 must be < 5°)

B_2 = <3 degrees



Z = 0.1 cm (Z must be < 0.32 cm)

W = <0.08 cm (W must be < 0.08 cm)

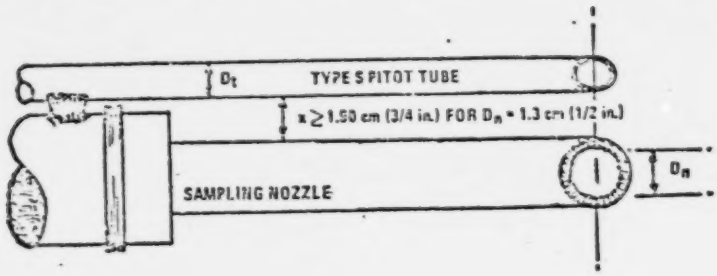
Data checked and approved for baseline coefficient (0.84)

By J. Howes Date 8/26/82

Figure C-3. Method 5 pitot tube calibration data (continued).

Pitot tube S/N (Integral part of Probe) Date 8/26/82

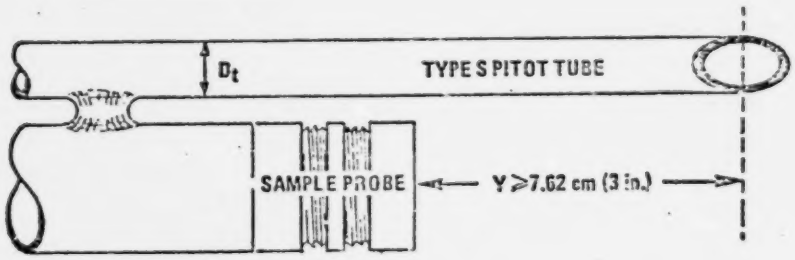
Probe S/N 569-44-1 Performed by J. Howes



D_t = 0.95 cm (D_t must be between 0.48 and 0.95 cm)

D_n = 0.77 cm (X must be ≥ 1.90 cm for D_n ≤ 1.3 cm)

X = 1.9 cm



Y = 8 cm (Y must be ≥ 7.62 cm)

Figure C-4. Method 5 sampling probe and pitot tube configuration.

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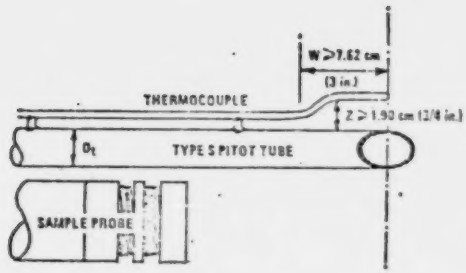
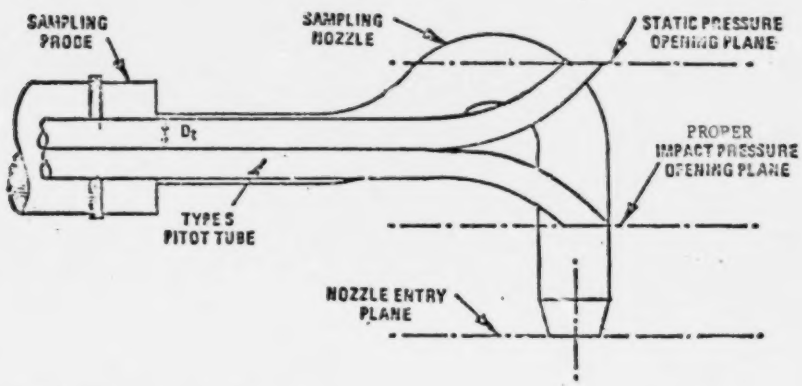


W = 8 cm (W must be ≥ 7.62 cm)

Z = 1.92 cm (Z must be ≥ 1.90 cm)

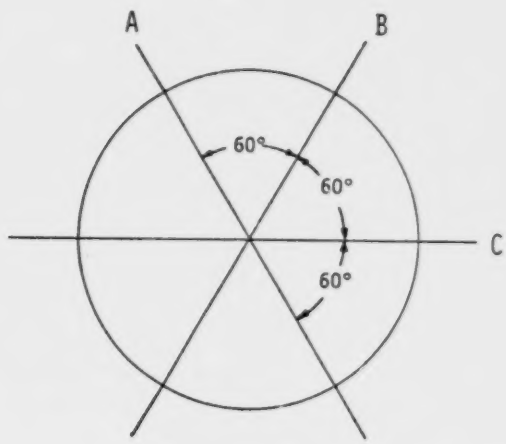


Impact pressure plane of pitot tube even with or above nozzle entry plane? (YES) NO (Circle)

Data checked by J. Howes Date 8/26/82

Figure C-4. Method 5 sampling probe and pitot tube configuration (continued).

Nozzle S/N 303-1 Date 8/28/82 Performed By J. Howes



Nozzle Inside Diameter Measurements (nearest 0.001 in.)

Diameter A 0.302 in.

Diameter B 0.304 in.

Diameter C 0.304 in.

Average Diameter 0.3033 in.

Figure C-5. Method 5 nozzle diameter calibration.

Calibration Date 9/8/82 Sample Box S/N BCL-546-1

Performed by R. N. Smith Dry Gas Meter S/N

Barometer (P_b) 29.55 in. Hg Wet Test Meter S/N AL-17-1-15912

Orifice Manometer, in. H_2O (ΔH)	Gas Volumes, ft^3		Temperatures, F				Time, min. θ	Ratio V_w/V_d, Y	Dry Test $\Delta H@$, in. H_2O
	Wet Test (V_w)	Dry Gas (V_d)	Wet Test (t_w)	Dry Gas Inlet	Dry Gas Outlet	Dry Gas Avg. (t_d)			
		I / F							
	20.008 ℓ / 0.7066 ft^3	I 1.605 / F 2.312	73.4		75/80	77.5	22.08	1.0070	
	20.020 ℓ / 0.7070 ft^3	I 2.495 / F 3.212	73.4		80/82	81	10.13	0.9999	
	20.010 ℓ / 0.7066 ft^3	I 3.658 / F 4.375	73.4		84/86	85	7.23	1.0069	
		I / F							
		I / F							
							Average	1.0046	

Figure C-6. Calibration data for dry gas meter used to sample natural gas supply.

APPENDIX D

QUALITY ASSURANCE AUDIT REPORT

The report on the on-site quality assurance audit of the program conducted by S-CUBED is presented in this appendix.

S-CUBED

ON-SITE QUALITY ASSURANCE AUDIT REPORT OF BATTELLE COLUMBUS LABORATORIES WORK ASSIGNMENT FOR:

"Determination of Dioxin Levels in Carbon Reactivation Process Effluent Streams"

SSS-R-83-5733
S-CUBED Ref. No.: 11183-03
S-CUBED Doc. No.: 0698V

FINAL REPORT

Task 3

Prepared by:
Bruce N. Colby and David R. Taylor

EPA Contract No. 68-03-3105

Project Officer: Mr. Lawrence Kamphake
Municipal Environmental Research Laboratory
Office of Research and Development
Washington, D.C. 20460

September 3, 1982

P. O. Box 1620, La Jolla, California 92038-1620
(619) 453-0060

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1.0 INTRODUCTION

The objective of this activity was to perform an on-site systems audit of EPA Contract No. 68-02-3487 Work Assignment 16 between U.S. Environmental Protection Agency/Municipal Environmental Research Laboratory (USEPA/MERL) and Battelle Columbus Laboratories (BCL). The audited work assignment was designed to determine the dioxin level in carbon reactivation process effluent streams at the Cincinnati Water Works. This audit report provides information regarding the degree to which quality assurance activities are being undertaken by BCL. The audit was designed to examine quality assurance activities during sampling at the Cincinnati Water Works (CW) and those procedures and facilities used in the Dioxin analysis at BCL. It should be noted that field samples had not been received by the laboratory at the time of the audit and consequently, the audit was confined to existing QA practices. It is assumed that these practices will be continued during analysis of CW samples.

Prior to the on-site audit, BCL's Work Plan and Quality Assurance Project Plan were studied to familiarize the auditors with project sampling analysis and QA activities. In addition, a Quality Assurance Pre-Audit Worksheet was sent to BCL. This worksheet was returned to S-CUBED prior to the audit and provided summary information concerning the organization, as well as QA, and sampling and analysis procedures being used by the contractor. Information from the worksheet was used to formulate questions prior to the on-site audit. The following sections of this report provide a summary with conclusions and recommended corrective actions (Section 2), brief description of the on-site audit (Section 3), and details of the on-site audit findings (Section 4).

2.0 SUMMARY, CONCLUSIONS AND RECOMMENDED CORRECTIVE ACTIONS

All aspects of QA seemed to be in very good condition and only a single minor corrective action is recommended. This is that the QAPP be updated to describe PCB analysis as it is currently planned for this project. It is also recommended that BCL make an effort to have QA personnel available during future audits. It might also be of value to BCL laboratory personnel to maintain a set of long term control charts for tetrachlorodibenzo-p-dioxin (TCDD) recovery and precision for common sample matrices such as H_2O, soil, fly ash and XAD-2. Although such charts are not necessarily significant on a per project/task basis, they could be useful 1) in establishing "ball park" estimates for precision and recovery, 2) in spotting "unusual" results, and 3) as a basis for monitoring improvements in analysis technique.

3.0 ON-SITE AUDIT DESCRIPTION

The on-site audit was performed on 1 September for the field sampling at the Cincinnati Water Works and on 2 September at Battelle's Columbus Laboratories for sample preparation and analysis. Both of the activities were performed by Dr. Bruce N. Colby of S-CUBED. Primary discussions in the field were primarily with Mr. J. E. Howes and in the lab, with Dr. F. L. DeRoos and Mr. D. G. Aichele. The BCL Project QAO was not available during the audit. Mr. Ben Lykins of USEPA/MERL was also present during the initial aspects of the field sampling portion of the audit. Project organization and personnel, facilities, sampling and analysis methods, quality control and data handling were reviewed.

4.0 DETAILED AUDIT FINDINGS

The following describes on-site audit findings in detail.

4.1 PROJECT ORGANIZATION AND PERSONNEL

Project organization and personnel appear appropriate for all aspects of this task. It is staffed with qualified individuals whose experience, training and backgrounds are consistent with project goals. It was unfortunate that the Project QA Officer, R. E. Heffelfinger, was not available during the audit; BCL should recognize that the availability of QA personnel during an audit is important and to try and prevent this situation from reoccurring in the future. Project personnel, however, seem highly aware of QA/QC and look upon it as an important aspect of sampling and analysis. The project schedule is reasonable and no technical problems should be anticipated due to timing complications.

4.2 FACILITIES

Laboratory facilities were found to be neat, well organized and provided with instrumentation appropriate for the project. The work areas appeared safe and contamination free. Handling procedures for TCDD seemed reasonable and appropriate. One minor recommendation, however, can be made, namely that calibration dates be attached to balances. This is common practice in many laboratories as is the posting of fume hood face velocities and check dates.

Major laboratory instrumentation for the project consists of a VG MM7070H mass spectrometer interfaced to a Carlo Erba 4160 capillary column gas chromatograph. This equipment, which will be used for TCDD and tetrachlorodibenzo furan (TCDF) analysis, is in excellent condition and quite capable of performing the analyses at hand. The

polychlorinated biphenyl (PCB) analyses, contrary to the QA Project Plan (QAPP), will be performed on a Hewlett-Packard 5830A packed column gas chromatograph with an electron capture detector. This instrument also appears to be in excellent condition. Operators manuals, calibration protocols and records, and service records were available for the instruments.

The Mass Spectrometry (MS) instrumentation had been modified to a minor extent. These modifications are consistent with the norm in high resolution GC/MS instrumentation and are done in such a way as not to adversely affect analysis results.

Sampling equipment, i.e., the modified Method 5 sample train, was in good condition and functioning properly during the audit. Field analysis equipment also appeared in good condition and to be functioning without difficulty.

4.3 ANALYTICAL METHODOLOGY

The analytical methodology for TCDD's and TCDF's appears completely appropriate for the project. There is a standard EPA method for TCDD, Method 613, but it is for water samples and this reviewer completely agrees with BCL personnel that the approach BCL will use for this project is superior to a modification of Method 613. This is true for two reasons 1) the $^{13}C_{12}$-TCDD reference will be added prior to sample preparation, and 2) high resolution mass spectrometry provides specificity essential for reliable part per trillion measurements.

The method for PCB's has not been documented for the application at hand. Laboratory personnel, however, appear fully knowledgeable of what they plan to do with this sample when they receive it. Although PCB's are not a major analysis parameter in this project, the intended methods should be documented. The recommended approach to this is to

update the QAPP which at present describes PCB analysis in very limited detail as a capillary column GC/MS (low resolution MS) method.

Written methods are available to the analysts who are currently producing neat and well documented bench records. Reagent quality appears appropriate; TCDD purity being evaluated via a round robin approach with other investigators. Standards are run on more than a daily basis. Detection limits seem appropriate for the task although insufficient health effects data are currently available for interpreting results. Samples are being handled properly and appropriate records are being maintained.

4.4 SAMPLING AND SAMPLE HANDLING

Sampling and sample handling are being performed in appropriate fashion. Sampling personnel, J. Howes and D. Kohler are both very experienced and knowledgeable in the use of the modified Method 5 sampling train and field sampling in general. Field records were complete, neat and recorded according to QAPP specifications. The sampling activity was well organized, proper containers were being used and they were labeled appropriately. In the strict sense, duplicate field samples were not planned for this project, however, the entire activity consists of quadruplicate sampling of a single process under constant conditions. Chain-of-custody records were available and were complete. Records were being maintained in bound notebooks. All samples are to be transported to BCL laboratories by BCL personnel in ice chests.

4.5 QUALITY CONTROL

Review of quality control activities was limited to past BCL TCDD studies. The TCDF's have not been investigated as frequently as TCDD's, and PCB's were not extensively addressed because of 1) lack

of method definition, and 2) their more minor project impact. Based on previous BCL quality control procedures for 2,3,7,8-TCDD, the laboratory and their methodology appears to be working well. Although they could not make any recent audit results available, they did indicate that the last check sample they analyzed resulted in approximately a -20 percent error. This seems more than reasonable for a performance evaluation sample in the part per trillion concentration range. Additional QC data were made available from a study performed for Dr. Larry Johnson and Mr. Merrill Jackson of EPA's Industrial Environmental Research Laboratory in Research Triangle Park, North Carolina, (IERL-RTP). These data indicated nominally 95 percent recovery of 2,3,7,8-TCDD from XAD-2 traps used to sample a bench scale "stack" at BCL.

No laboratory QC manual was available at BCL, but all the items normally addressed in a Laboratory QC Manual are addressed in the QAPP. Consequently, this is not considered a problem.

4.6 DATA HANDLING

Data handling procedures appear appropriate for the project and well under control. No statistical tests are applied in reviewing data but, owing to limited QC data on any specific matrix this seems reasonable. Data are subjectively reviewed for "anomalous results" by laboratory and project personnel who seem quite capable of picking up outliers based on experience. It might still be of value, however, for BCL to maintain and chart long term QC results for precision and recovery on a nominal matrix basis. This would provide 1) a means for estimating precision and recovery, 2) a data base from which to spot outliers, and 3) a mechanism for monitoring long term improvements in analytical technique.